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PUBLISHED BY THE TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY

SEPTEMBER 1959

VOLUME 42 • NUMBER 9

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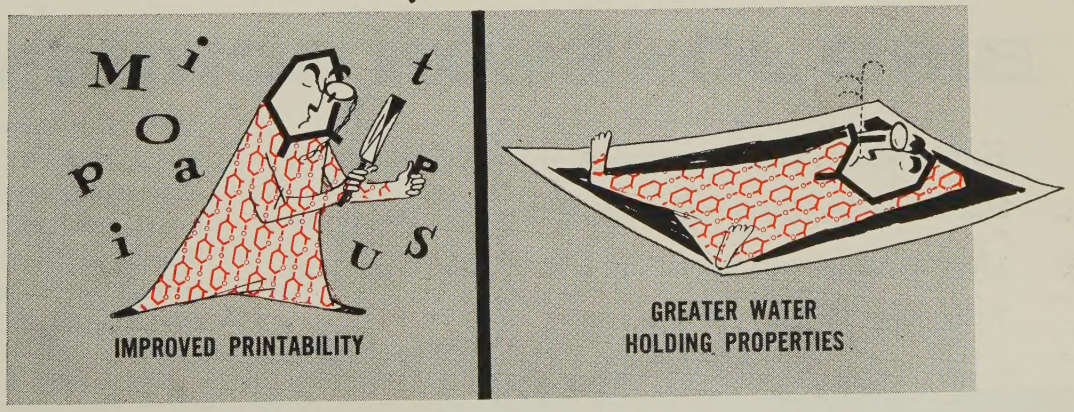

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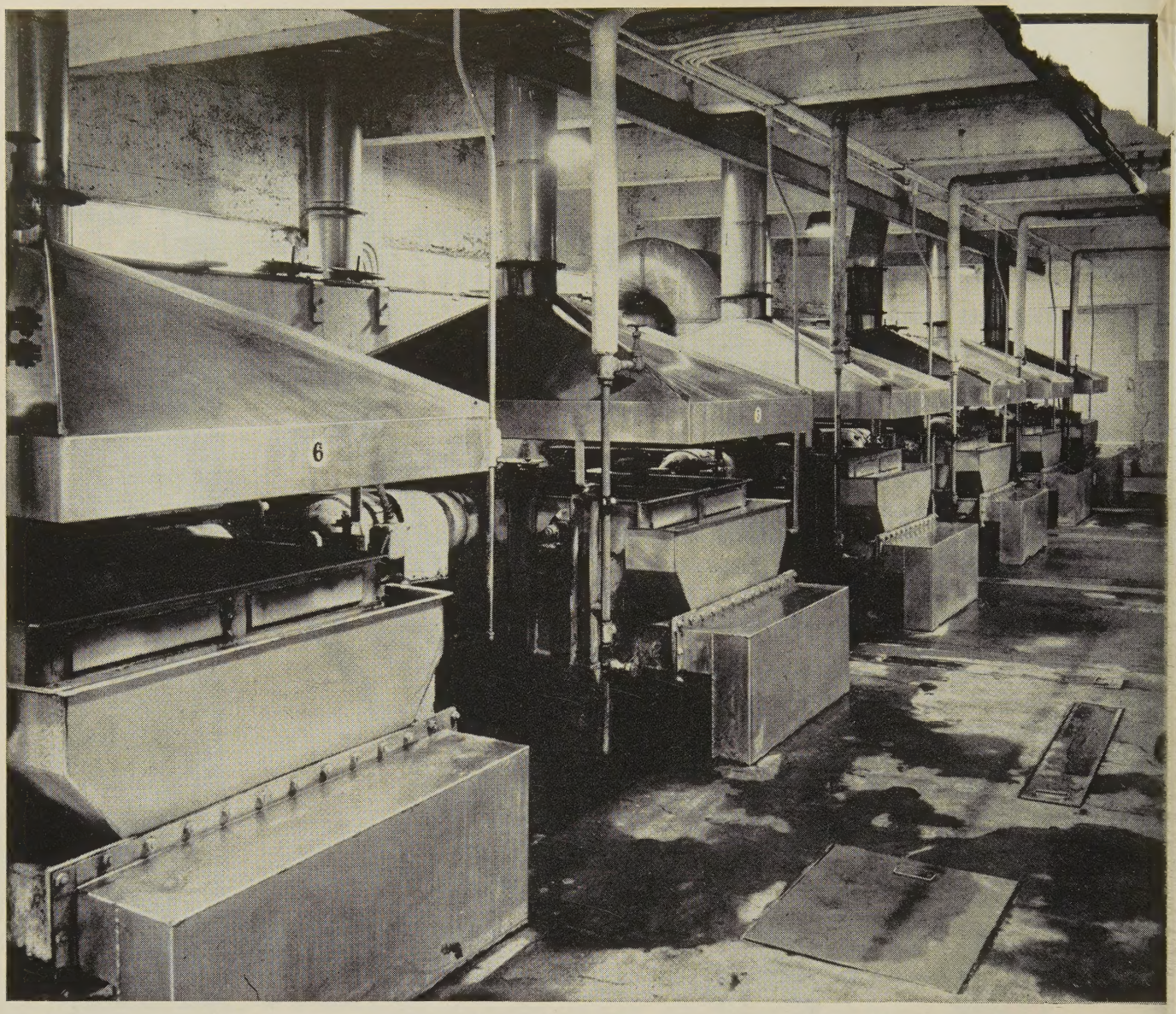

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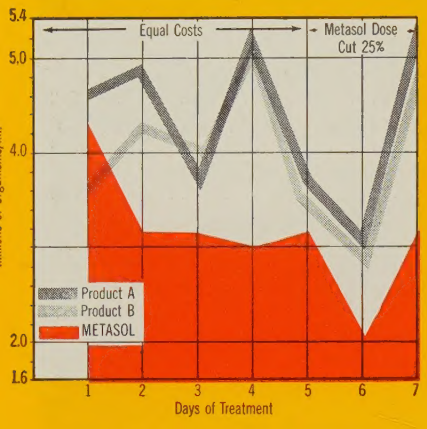
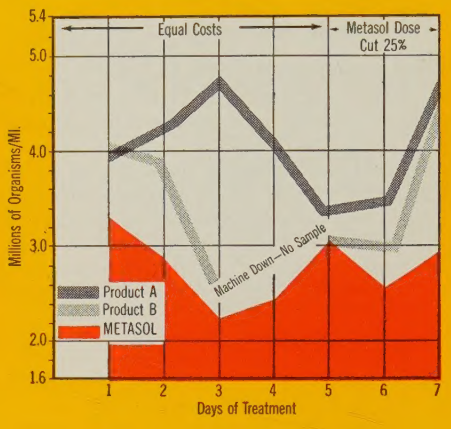


25% to 35% MORE EFFECTIVE QUICK KILL

Daily count of organisms per milliliter over a seven day period, **one hour** after addition of slimicide.



30% to 40% MORE EFFECTIVE SUSTAINED KILL

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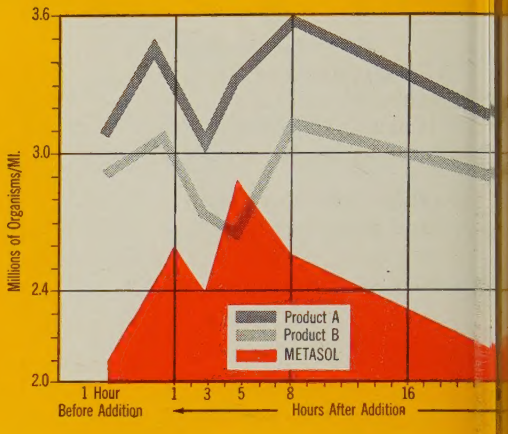

25% to 30% MORE EFFECTIVE AVERAGE KILL

Daily count per milliliter on hourly basis, from one hour before addition of slimicide to 23 hours after

The same conclusive result from tests at three leading mills

METASOL compared with conventional slimicide

In this bleached-kraft, hardwood pulp mill, solid METASOL replaced a solid conventional slimicide on an equal cost basis. Schedule for both slimicides was three times a day, at eight-hour intervals. All exposed parts, both wood and stainless steel, had been slime-coated when previous slimicide had been used. METASOL cleaned them after the first run.

Compared with a second machine, in which no slimicide was used, the METASOL-treated machine showed an organism count only half as high, even though the no-slimicide machine was washed down 7 to 15 times as frequently as the METASOL unit. The low count was maintained throughout six months of continuous use.

METASOL in ground wood

METASOL was tested against two conventional slimicides in a newsprint mill using a mixture of ground wood and sulphite pulp. All three were run parallel at equal prices.

After only a few days, METASOL showed a far superior level of protection. At the request of mill management, the METASOL dose was dropped 25% in cost below the other two slimicides. METASOL continued to show a superior rate of protection and, over a two-week period, built up still more protection, then leveled off evenly. Over a three-week period, the machine using METASOL had higher productivity, no sign of microbiological slime break, and a much lower organism level.

METASOL in comparative performance test

In a test at a bleached-kraft mill one machine was treated with solid METASOL and the other with a solid conventional slimicide on an equal cost basis. At the start of the test, both machines showed a count of 4,000 organisms per milliliter. After 24 hours, the machine using the conventional slimicide showed a slime build up. Consequently, the METASOL treatment was reduced one-third.

At the end of a week, the count on the machine using METASOL had been cut to 2,000 per milliliter, while the count on the other machine had built up to 12,000 organisms per milliliter. In addition, the machine treated with METASOL had produced 17% more paper.

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horoughly tested by leading mills, METASOL has en found superior to any other slimicide on the mar- t. Every mill which has tried it has decided to use it a continuing basis.

Keeps machines running

ETASOL lowers production costs because it keeps achines clean and helps them deliver a finer product ithout loss of time. One mill which previously had to ut down weekends for wash-downs now runs five con- nuous weeks without a shut down. Daily production as gone up also.

Protects against all organisms

f all commercial products tested, this new slimicide rovides the most effective "quick kill" and "sustained ill" action against bacteria, fungi and other micro- rganisms. It is so effective that doses can be reduced ubstantially compared with conventional slimicides, and still produce better results. Furthermore, tests show that, unlike other slimicides, METASOL does *not* permit microorganisms to develop resistance.

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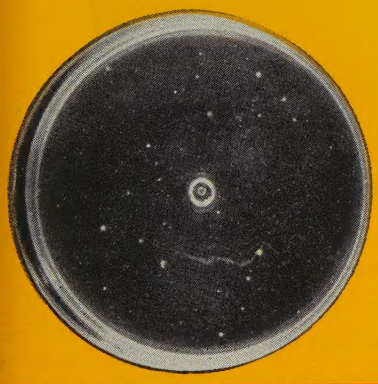
Demonstration in your mill

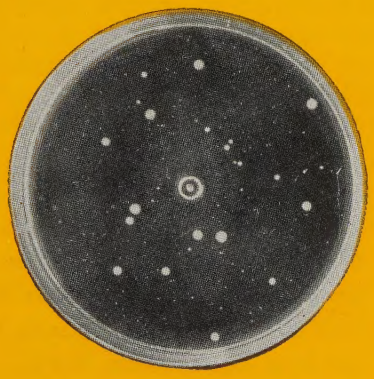
METASOL chemists and micro-biologists will demonstrate its effectiveness in your mill, on your machines, under normal production conditions. On request, they will analyze your particular slime problem, recommend corrective and preventive measures, and train members of your own organization in efficient slime-control methods.

Use the coupon on the bottom of this page to ask for your copy of a new brochure which shows exactly how METASOL slimicide works, and actual comparative performance records under mill-run conditions.

21 DAY TEST SHOWS SUSTAINED KILL



METASOL



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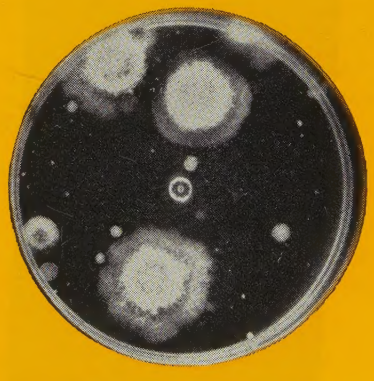

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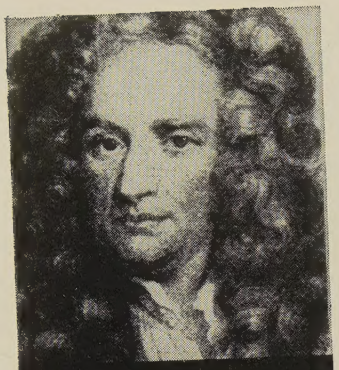
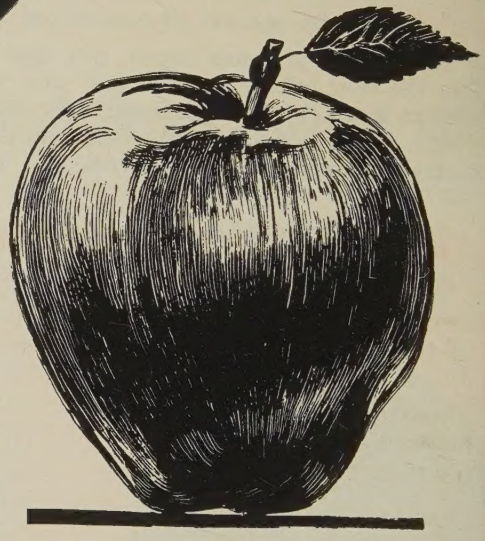


Isaac Newton

$f = Gm_1m_2/r^2$



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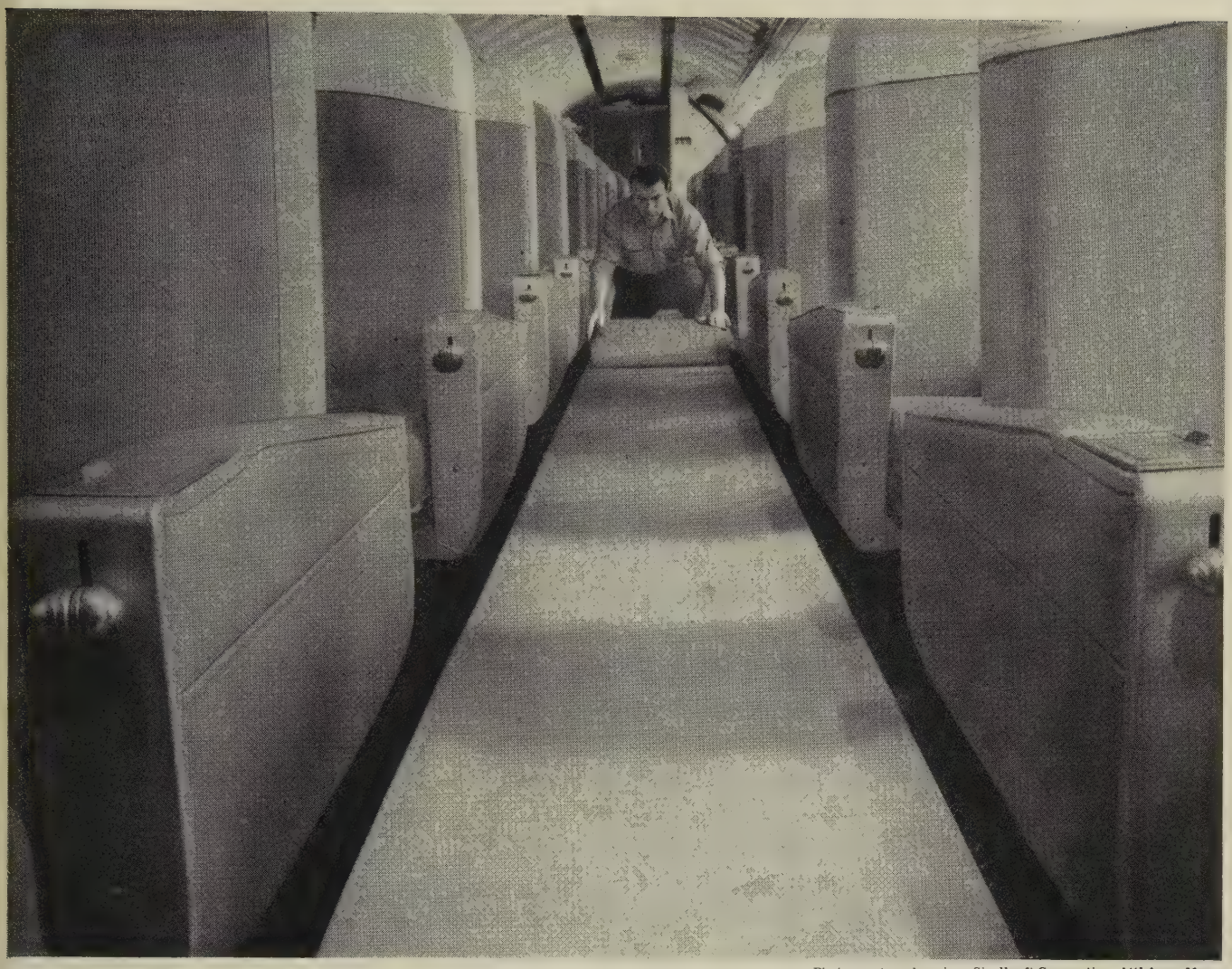

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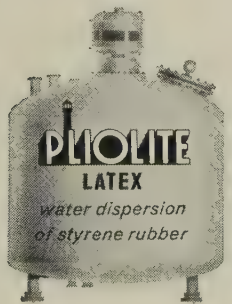

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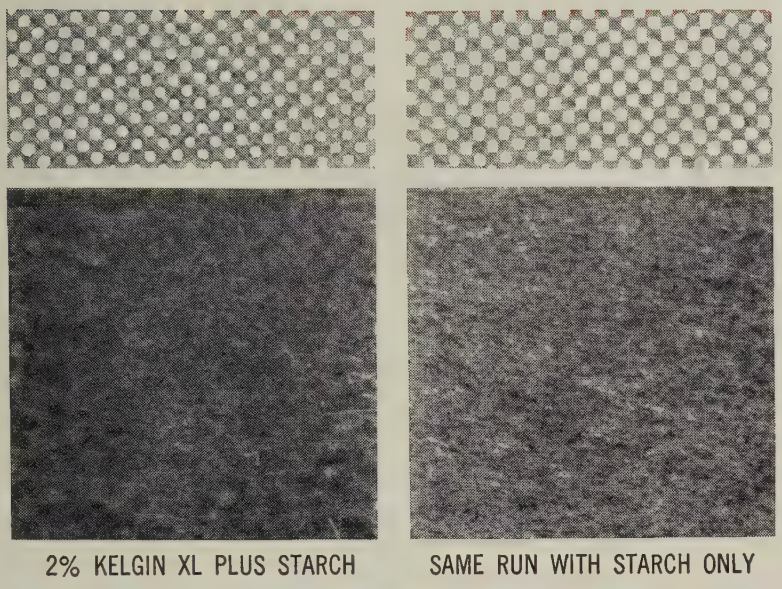

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7168

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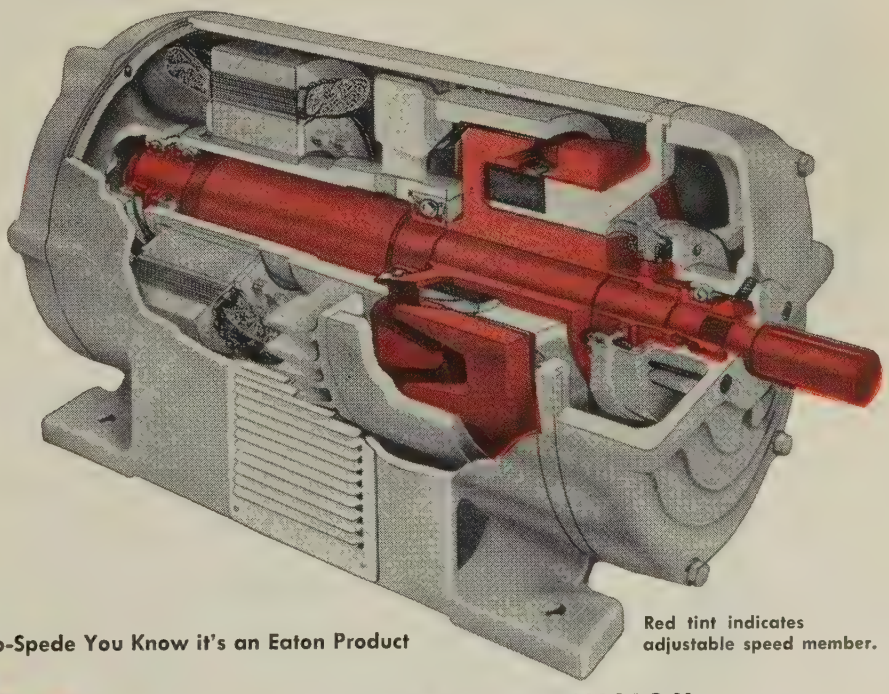

Red tint indicates adjustable speed member.

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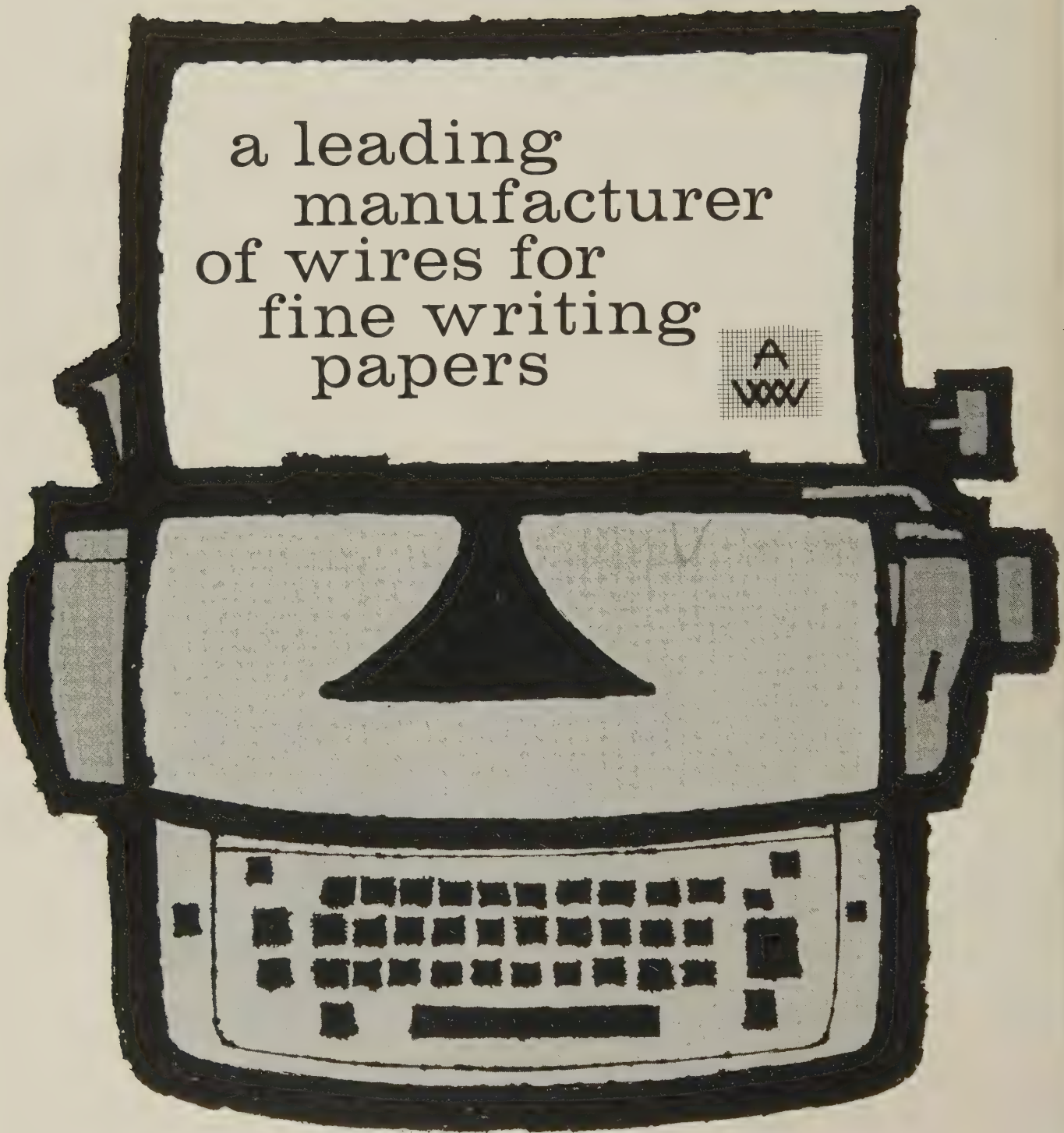

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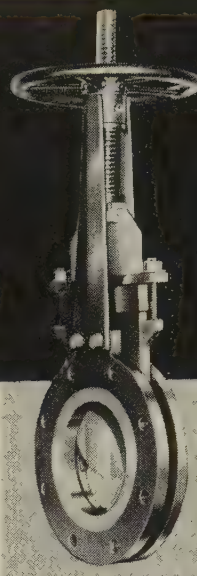

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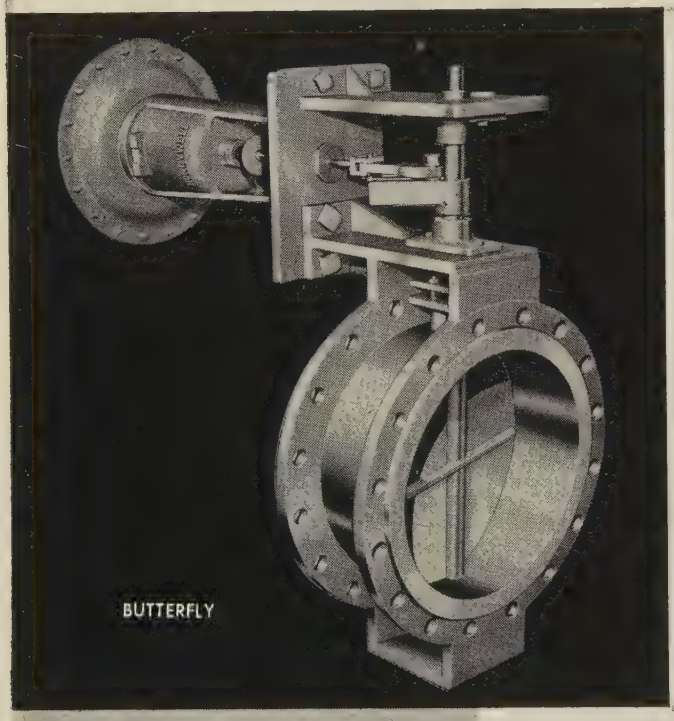

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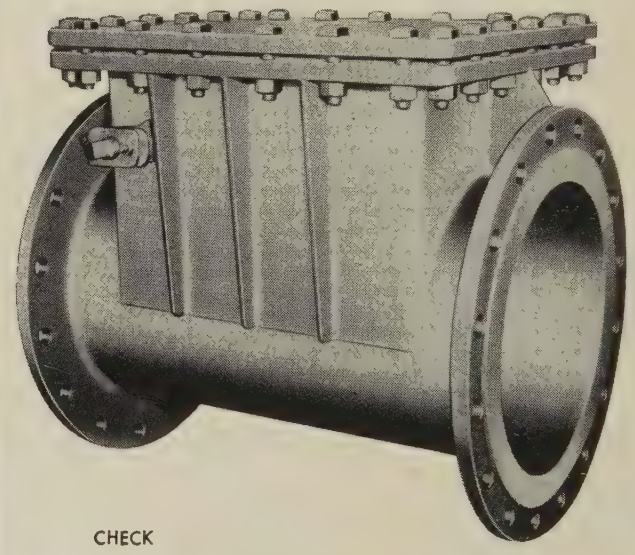

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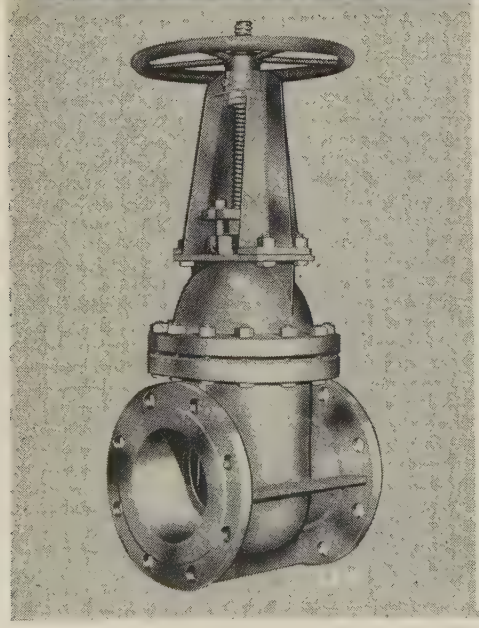

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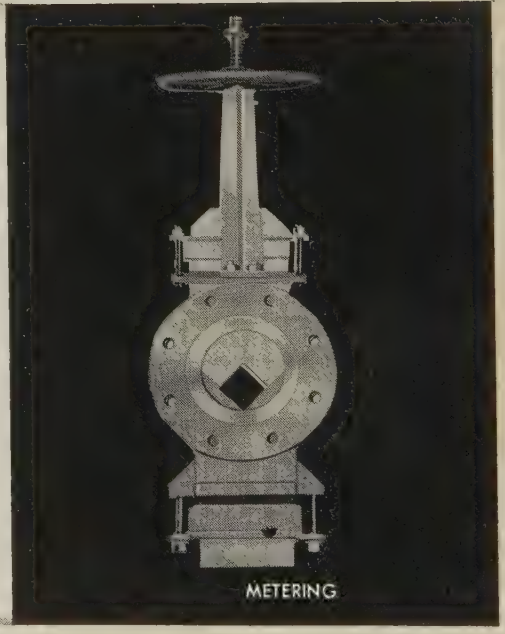

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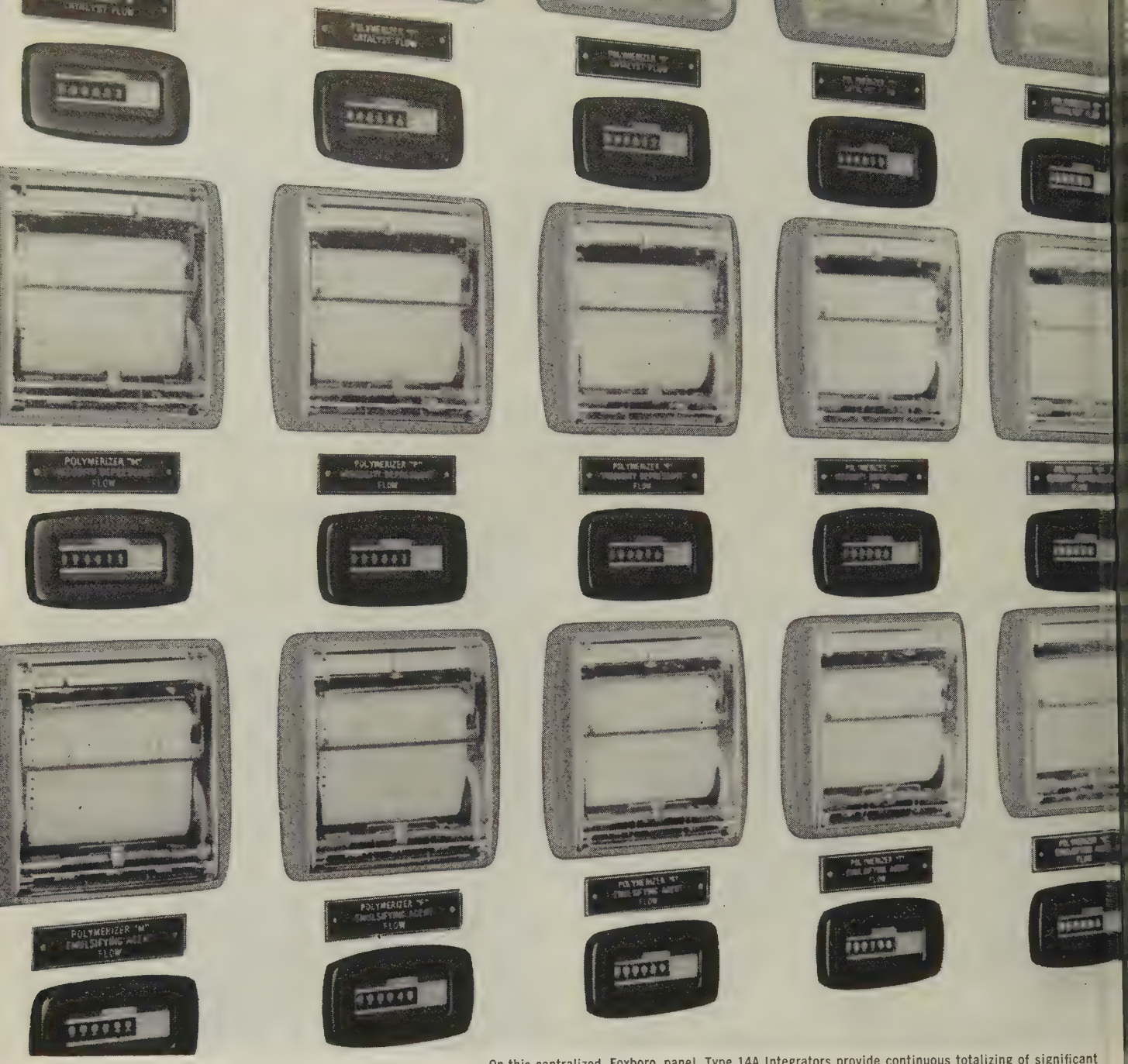

On this centralized Foxboro panel, Type 14A Integrators provide continuous totalizing of significant process and plant service flows – recorders give trend records required by operating personnel.

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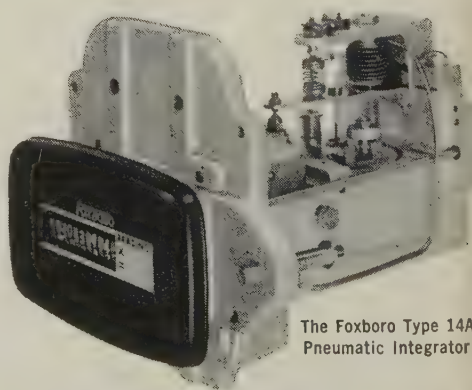

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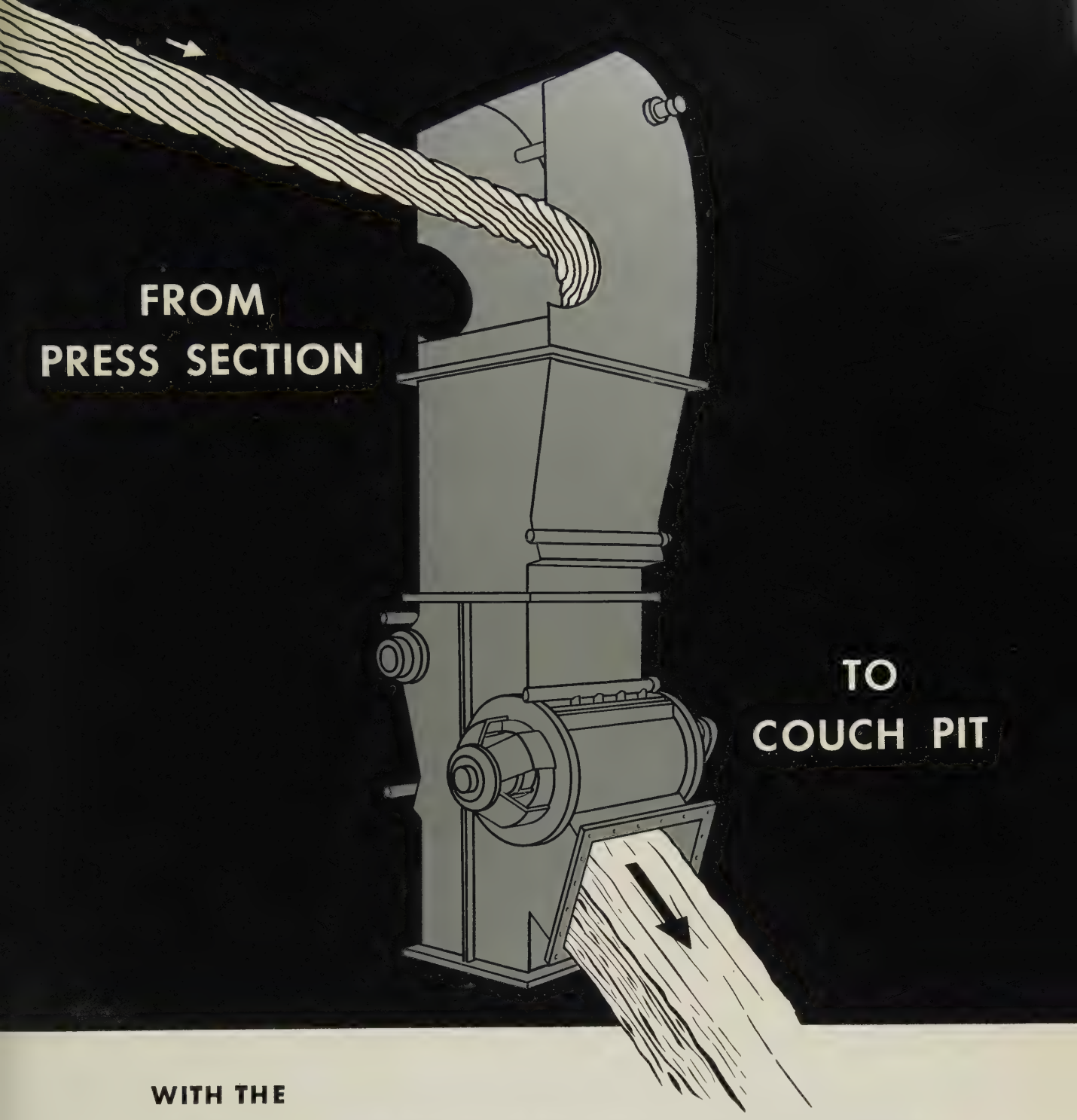

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Viscosity (Brookfield at 25°)	150 ± 50
Mechanical Stability	Excellent

Physical Properties

Specific Gravity	1.01
Weight/gal.	8.43 pounds
Refractive index (D^{25C})	1.530
Film properties	Clear and glossy
Adhesion	Excellent
Particle size	900 Angstroms 0.09 Microns

GEN-FLO® 62 styrene-butadiene latex for casein or starch coating formulations

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TSC, % by weight	48 ± 1.0
pH	10 ± 1
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Brookfield Viscosity, cps	15-25
Mechanical Stability	0.1 max 100 mesh screen

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Trees and Game—Twin Crops*

ARTHUR H. CARHART

TREES and wildlife live together on nearly half a billion acres of commercial timberlands in the United States. A great many of our native animals and birds require forest environments for food and shelter.

The values of natural resources lies in people making wise use of such wealth. Therefore, wise management of timber and of game is directed at the production and harvest of each as a crop—indeed, they are twin crops.

Well-planned management of our timberlands means two things of prime interest to sportsmen. First, in most instances, it assures high levels of production of both wood and wildlife. Second, it is the best prospect we have of sustained yields in both resources.

In this discussion we explore the coexistence of trees and animals; how animals live in and near our timberlands, mostly in congenial association with trees, sometimes in conflict.

It is also our purpose to show how people interested in timber and those interested in wildlife can assist each other in producing and harvesting more timber and more game from wooded acres.

FOREST IS A CHANGING COMPLEX

We must keep in mind that a forest is a constantly changing complex made up of bits and quantities of many things. Even a minor shift of the status of any part may affect numerous other segments. A major change, such as harvesting a crop of game animals, or ready-for-use trees, can cause severe impacts on other forest values. Such effects may be adverse or distinctly beneficial.

Moose, elk, deer, bighorn, sheep, mountain goats, and bears are timberland dwellers during some stages of their existence or in certain seasons. These animals would be hard put to survive if forested acres suited to their needs were unavailable.

Woods are homeland for many of the furbearers. Grouse, partridge, woodcock, and other games birds find homes in or near timber. Small game such as rabbits, woodchucks, all varieties of squirrels, locate their dens and burrows in or close to the woodlands.

Trees are the fisherman's best friends because the managed forest excels in protecting slopes that gather the waters of streams. Wooded acres assure more constant stream flow and water of better quality. Tree growing lands prevent excessive and destructive erosion—the silt which is the greatest by volume of all water pollutants.

Another contribution to underwriting wildlife values is the protection timber-covered slopes give the habitat of waterfowl. Silt carried by floods from unmanaged or poorly managed watershed lands is most certain death for pond, slough, or marsh. Timbered hillsides do not bleed silt.

For such reasons, approximately 40 million hunters and fishermen who each year buy licenses, plus 20 million youngsters and others privileged to hunt and fish without a license, have the keenest interest in how our forest lands are managed. The student, naturalist, and vacationer also have their stake in how forests are handled. Having lands dedicated to growing trees is the most positive assurance we may have that our forest wildlife will continue to live in natural homelands rather than to exist as remnants preserved in museum-like parks and zoos.

Let's now explore and discuss how good forest management and the management of many types of North American wildlife are linked together.

WILDLIFE MANAGEMENT IS YOUNG

The profession of wildlife management is less than a quarter century old. Acceptance of its position in conservation has come only within the last decade or so. The present-day systematic approach to wildlife's needs arrived after and partly because of numerous fumbles, flops, and calamities in game and fish administrations.

As the year 1900 arrived, many kinds of American wildlife faced serious trouble. Bison, elk, and woodland caribou had been wiped out in eastern states. Passenger pigeons and heath hens were entering the realm of legend. In numerous localities once-abundant deer approached oblivion.

In Michigan there was another story. Here, briefly is what happened there between 1880 and 1900.

Old trees that cast dense shade had been logged off. Unopposed wildfire swept away cull and weed trees. Brush and young tree sprouts sprang up. Thousands of acres became loaded with excellent deer forage.

Wherever nature produces an extravagant supply of food, wildlife populations often "erupt." The Michigan deer population erupted. At the high point considerably over a million head of deer fed through thickets on 8 million acres.

No exact data were kept of this population eruption. Field evidence and news stories of the day indicate that the rapidly multiplying deer herds ate themselves out of food supplies. Surviving trees grew so tall that tender tips stood above the reach of hungry deer. A new forest canopy formed. Shrubby browse plants not eaten out by deer were shaded out by new forest.

Uncounted hundreds of thousands of Michigan deer died.

Market hunters were generally blamed for the collapse of Michigan deer herds. Early efforts at saving wildlife centered at halting overshooting. Closed seasons and increased law enforcement had their values, but they were no cure for such a situation as that affecting Michigan deer from 1880 to 1900.

So a new movement, another type of protection, began about 1900. Game refuges and sanctuaries were hailed as the panacea for saving our wildlife.

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The Witchita National Wildlife Refuge in Oklahoma was established in 1905. The Lower Klamath National Wildlife Refuge, a vital link in the Pacific Coast flyway used by migratory waterfowl, was made official in 1908. The famous National Elk Refuge in Jackson Hole, Wyo., dates from 1912. The Charles Sheldon Antelope Refuge in northwestern Nevada was closed to hunting in January, 1931.

States began throwing big acreages into bigger, and it was believed, better, refuges. Some were so large a big game animal might live its entire life within refuge boundaries. Such a long, protected life could be devoted mainly to eating and producing more of its kind.

A widespread campaign of trapping, shooting, and poisoning predatory animals began about 1918. That removed some of nature's checks on many game species and allowed populations to spiral upward.

Oversize refuges began to be incubators of wildlife catastrophes.

CASE OF KAIBAB DEER

Harold Titus, the distinguished conservationist who wrote "Timber and Game—Twin Crops," the antecedent of this book, ably reported two important milestones of wildlife management reached soon after World War I.

The first milestone was "The Case of the Kaibab Deer." This is what happened to those deer:

The Kaibab Plateau is a vast tableland north of the Grand Canyon in Arizona, much of it within the Kaibab National Forest. The great canyon lies as a barrier against deer traveling southward. On the other three sides is desert. Kaibab deer were, and are, dependent on what that plateau offers.

Mature ponderosa pines in moderately open stands were typical trees of the Kaibab. Cliff rose, aspen sprouts, and twigs and shoots the deer could reach provided fairly abundant browse beneath the high-crowned evergreens. Both the deer hunting and timber were "hard to get to." The harvesting of deer by hunting, of timber by logging, might have prevented calamity. In their isolation the populations of Kaibab deer herds began to pyramid.

Between 1918 and 1924 the herds built from an estimated 20,000 head to over 100,000. The original food supply could have maintained only about 32,000 head of deer.

Browse was eaten out. Deer died in droves. Men argued over authority to permit emergency hunting to remove surplus animals and save the battered deer range. Several highly qualified observers believe the Kaibab deer population hit bottom with less than 5000 surviving.

The Kaibab story brought certain facts into sharp focus. First, with adequate checks removed, game populations starting in good environment often erupt, then skid to near-extinction. Second, the Kaibab record was a positive demonstration of how management plans for every part of the forest complex are inseparably intertwined.

The seond milestone to which Titus directed attention was a study of the bobwhite quail, made by Herbert L. Stoddard in Georgia between 1924 and 1928.

Pioneer hunters undoubtedly knew that forests solidly blocked with mature trees are not good game territory. The part of a forest preferred by wildlife is the "edge." As Dr. Ira N. Garbrielson stated in his book, "Wildlife Conservation," a virgin forest is practically a biological desert. The "edge" means not only the forest border, but it includes openings and clearings within the forest.

Stoddard's studies brought sharp attention to bear on the high value of the forest "edge" as game habitat. It was, in a considerable degree, confirmation of what had been demonstrated in the Kaibab—for it was abundant brushy browse growing in open pine and aspen stands that first underwrote the upsurge of deer populations. Then, as it was eaten out, its disappearance sealed the doom of the deer.

Prior to Stoddard's work, technical studies of wildlife often were made by vacationing university biology professors. Or naturalists studied wildlife in less exact ways known as nature study. These scholars contributed greatly to wildlife work that was to follow, but generally they lacked opportunities and funds to do the job that had to be done. State game and fish departments needed facilities to dig, and dig, and dig, deep, with thoroughness and twelve months each year, for vital *facts* concerning wildlife needs.

The needed funds became available to the states in the late 1930's.

The 11% excise tax on sporting arms and ammunition that had been collected under the Federal Aid in Wildlife (Pittman-Robertson) Act, was first put to work in the autumn of 1938. Among the activities approved by the law and the supervising U. S. Fish and Wildlife Service was a program of systematic studies of problems affecting game species. Later the Dingell-Johnson Act supplied the same opportunity with relation to game fishes and sport fishing. The states now can put trained men into the field to procure the facts essential in sound management of game species.

World War II temporarily derailed the Pittman-Robertson program. Since the war, hundreds of trained young men have assembled more information about American wildlife than was recorded from Colonial times to 1940. Encounter these "college dudes" in the field and you'll probably find them in jeans, dusty, muddy, whiskery, and deep-plunged in their exacting, often exciting work. Don't sell them short; they know their business. They are as fully qualified in their field as the young forester is in his work. Teamed with the forester, they can assure results in managing "Trees and Game—Twin Crops."

This is the third major milestone, the one of paramount importance that marks the arrival of modern wildlife management. Most of the former guess-and-hope techniques by which officials did their best to conserve our wildlife has been replaced by knowledge, that most potent of all management tools.

Let us now take a brief inventory of the lands of the country that are classified as "forest." These are prime game producing areas on which foresters and game managers may work together to assure future crops of trees and wildlife.

FORESTS PRODUCE WOOD AND WILDLIFE

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represent the size of our forests. The acres they tally are fields from which we will harvest corps of wood and wildlife.

Look beyond the figures. You will then see the woodlots, groves, great blocks of man-planted forest growing rampant, the millions of other acres of naturally reproduced forest. Then these figures may translate into something as big as all outdoors.

Before Europeans arrived on this continent, about 822,000,000 acres of what now is the United States were forested. More than a fifth of this acreage has been cleared of timber and serves other needful uses—in farms, cities, giant reservoirs, highways, airfields. We have 648,000,000 acres that should be maintained as forest.

Foresters classify some 484,000,000 acres as "commercial," that is, timberlands which produce timber in commercial quantity for sale and manufacture. "Commercial" does not refer to ownership status.

The largest block of commercial forest land under one management is in the national forests, 81,314,000 acres. Approximately 15,000,000 acres of reserved forest land are in national parks and similarly reserved federal areas where, for the most part, neither logging nor hunting is permitted.

About 28,000,000 acres in western grazing districts are classified as forest land.

States own 19,000,000 acres of commercial forest, and 8,000,000 acres are owned by counties, towns and villages.

These are the government forests. They contain a lot of acreage.

But here are figures more arresting.

Of the 648,000,000 acres of forest in the United States, 358,250,000 acres are *privately owned.*

Ownership of this vast acreage is spread among 4,510,000 individuals or companies.

The average size of these private forest properties is 79 acres.

About 300 owners hold title to holdings of 50,000 or more acres each. The average size of these "large" properties is 127,317 acres. The owners are timber companies, pulp and paper enterprises, mining, railroad, oil, power, water system, and other concerns. They are known as "industry forests."

Subtract 300 "large owners" from the 4,510,000 who each and severally own a chunk of forest, and you still have most of the 4,510,000 who hold title to three-fifths of the commercial forest land in the nation.

Taking 500 acres as the upper limit of forest holdings to be regarded as "small," the average across the nation is 49 acres per owner.

With varying degrees of success, the agencies responsible for government forests and state-owned wildlife have cooperated. We should assume these government agencies will do their best to achieve good management of the resources in their charge.

It doesn't take much figuring to reach the arresting conclusion that some of the most hopeful prospects for producing a greater abundance of wildlife are in the privately owned forests.

It should be equally clear that these acres will greatly benefit wildlife if they are assigned to and maintained in wood production. In fact, if we can find the way toward having most of the $4^1/_2$ million owners of private forests adopt good timber management practices *we have a great potential increase of the most productive type of wildlife habitat in the nation.*

Question is—how might this gigantic job be done?

THE TREE FARM PROGRAM

Though still in its infancy, the nation-wide movement initiated by the wood-using industries, known as "tree farming," gives promise of bringing many smaller forest properties under good timberland management. Most of the larger holdings are accredited Tree Farms. Thousands of the smaller ones also are enrolled in this tree growing movement.

Those who launched the Tree Farm program agree that their formost interest in it is based on long-range business planning. A number of timber and pulp companies own sizable blocks of forest land, but few have enough wood production potential on their own lands to keep mills and plants running indefinitely.

Taking a hard look at the future, these companies realize they must have greater supplies of wood than can be harvested continuously from their properties. The smaller properties include 62% of all commercial forests. A high level of production from all these must be achieved if these forest products companies are to have a future wood supply. The consumer, meaning you also, has a stake in having those smaller holdings growing wood at full capacity; you're the one who will feel the jolt of higher and higher prices for wood products if we fail to get most of these acres under full-scale management.

And remember, increased wildlife production usually results from good forest management.

To get most of these acres to growing all the wood possible is a Herculean task. This program is a challenge to every forester, sportsman-conservationist and woodland owner.

Tree farming in itself is not an involved operation. It means handling a plot of land to grow good trees so they may be harvested as a crop, again and again. Whether a Tree Farm contains 100,000 acres in Georgia or is a 20-acre woodlot in Wisconsin owned by a fellow named Peterson, Pottelby, or Pavich, its primary job is to grow useful wood. But as it is actively busy at achieving that no. 1 purpose, it also protects watershed, grows wildlife, cloaks hillsides with mantles of greenery, and may supply recreation such as only woodlands may afford.

This is not the place to state in detail how a private property, suited to growing wood, becomes a unit in the nationwide Tree Farm system. Briefly, these are the general requirements.

The property must be privately owned, tax-paying, and dedicated by its owner to growing and harvesting repeated crops of timber.

The forest must be managed in accord with good practices.

Protection against fire, insects, disease, and destructive grazing must be in basic management programs. Acceptable protective measures may include such items as constructing fire lanes, removing dead trees and other fire hazards, and having equipment ready for fire fighting. Fire lanes and access roads both can provide forest "edge" so important to wildlife.

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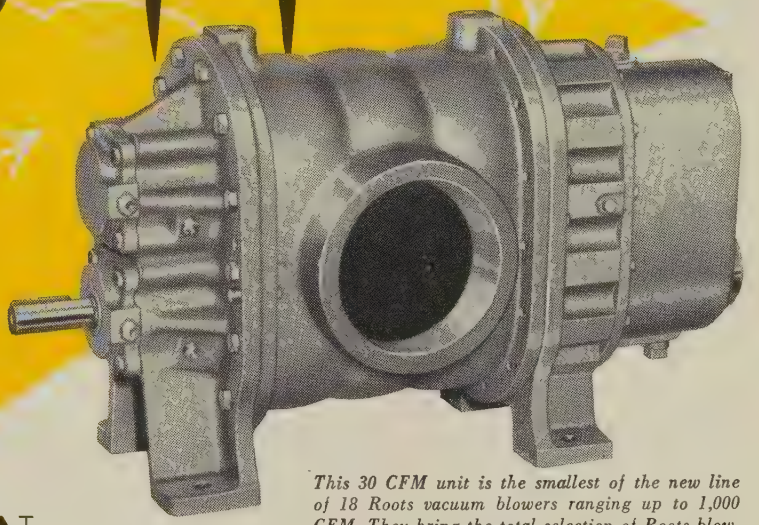

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Your nearest Roots-Connersville sales engineer has full details on this new series of vacuum blowers. Or write for a specification sheet and for Bulletin VP-158 covering the larger units.

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vesting of trees, the accredited tree farmer gives consideration to other inherent values and uses such as watershed protection, wildlife, and recreation.

Since the property is managed to produce repeated crops of trees, harvest by accepted methods is part of the standards applying to the approved Tree Farm. Furthermore, each harvest must provide for establishing the next crop of trees.

Certification of property as a Tree Farm usually is the job of a state-wide Tree Farm committee. Full information on steps to be taken can be secured from the American Forest Products Industries, 1816 N Street, N. W., Washington 6, D. C.

You probably are fully aware of how much more privately owned forests contribute to greater crops of wildlife and game, as well as timber, when managed as Tree Farms. Now comes your logical question.

"Suppose sportsmen-conservationists back the Tree Farm program—what about 'No Trespass' signs? What's the chance of my hunting partner and me getting any more hunting if there are more of these Tree Farms?"

The most assuring answer to that question is case after case of Tree Farm forests, where true sportsmen are welcome.

HUNTERS PLEDGE FOREST PROTECTION

Each season a paper company in Pennsylvania has issued approximately 1000 permits to hunt on its large Tree Farm, which is heavily populated with deer. In receiving the permit the hunter pledges himself not to build fires on the forest lands, nor damage property. He also pledges he will help fight forest fires if they break out, prevent damage-doing by others if he can, and report to the company as a good and cooperative visitor any off-trial doings he may come across on that Tree Farm.

"Welcome Hunter" signs greet sportsmen entering the 275,000 acres opened to their use (except some outright sanctuary areas), by a paper company in the South. This company, a large one, instructs crew workers to leave persimmon, hickory, cedar, and mulberry trees as food-producers for game.

This company also floods pin oak flats for winter feeding of ducks; the flats are drained each spring by breaking the dams so no lasting damage is done to trees.

Two oil companies in Louisiana welcome hunters to their holdings, while another forest industry not only welcomes sportsmen but is cooperating with Louisiana State University's wildlife studies by having fenced an area of 4500 acres assigned to this use.

In North Carolina still another pattern shows up in cooperation between the State Game Department and a paper company. As the company makes a planting of small trees after clear-cut harvesting, the state comes in and sows seed which produce superquail cover. Until pine overtop the lesser plants, hunters enjoy the finest sort of quail hunting for they can shoot over tops of the small pines.

In Florida the same company has tied in with state and federal agencies and with other industries in wildlife management on 5 million acres. To underwrite the cooperative program, a moderate fee in addition to state licenses is collected from hunters using the area.

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In the Pacific Northwest a large lumber and pulp company not only opened one of its Tree Farms to hunters, but supplied maps of the unit, gave sportsmen hot coffee, and had visitors sign the "Red Hat Pledge" of the Izaak Walton League which puts in words basic creeds of real sportsmen. With this positive approach, it was reported soon after season closed that:

Among 500 hunters there were no civil nor game laws violated.

No lost hunters were reported.

No uncontrolled fires started during hunting season.

No gates were broken down, no equipment stolen or damaged.

Signs were respected and sportsmen kept out of dangerous areas where the company had asked there be no auto travel.

In Maine one company cooperates with the state to protect and improve spawning runs of Atlantic salmon and alewives in streams within its properties.

Another Maine company teams up with the state to develop wildlife habitat, even to a limited use of heavy equipment in the cooperative program.

These cases are representative of how the leaders in the Tree Farm program have produced and provided for the harvesting of timber crops and game. A recent survey of 46,263,852 acres of privately owned forest land revealed a million and a half visitors enjoying these lands, including those coming to hunt and fish. The survey was limited to 455 companies owning "industry" forests. Of the properties on which reports were secured, hunting was provided for on 42,737,538 acres which is 92.4% of the total. Nearly 900,000 acres were in cooperative game refuges and management areas, and 31 companies employed game specialists.

Near where you live there may be timberlands owned by your neighbors that could qualify as a certified Tree Farm—which is another term for sound forestry practices that inevitably will underwrite more productive wildlife management.

Small, medium, or large, whatever the acreage of a block of timberland may be, there's a wide open opportunity for the hometown sportsmen to promote its establishment as a Tree Farm.

Furthermore, the sportsman-hunter has a vital function in basic operations of tree farming. And here is why he has the function!

CONSERVATION'S GOAL—HARVEST FOR USE

Basically, tree farmers and informed sportsmen are genuine conservationists. They believe in the same goals. A primary precept of both is prevention of waste in natural wealth. To reach its proper objective, natural wealth must be used; it must perform a service.

With this philosophy, the tree farmer would wish to have put to use the game, fish, and recreation available on his timberlands. For this is a way of thinking that is a tie between all who may be regarded as the real article in conservationists.

But there is a far more definable set of reasons for the sportsman and tree farmer to cooperate. Several woodland creatures are tree eaters. If a species starts pyramiding in population, headed toward ultimate disaster for itself and the food supply on which it exists, the one most feasible means for checking that eruption is a well-thought-out hunting harvest. It can be, in an extreme situation, as imperative to reduce overabundant animals as it is to fight a forest fire.

Porcupine, elk, bear, and beaver feed on tree parts in sufficient amounts to greatly damage or destroy forest growth. Topping all in this category are deer.

Since the turn of the century, deer herds have staged a phenomenal comeback. Practically every community with even a little forest cover nearby has its deer herd—and often its deer problems.

Deer are our most nearly universal big game. As such their management is of concern to most sportsmen, most tree farmers. These are illustrative of management problems on which forester, sportsmen and game and fish officials may, and need to, apply fullest cooperation.

To assure this cooperation, many of us must discard certain long-nutured misbeliefs and myths about deer. Regardless of much data we now have, some of these unfounded beliefs persist and have raised hob with the best management of timber and deer.

The principal portion of deer diets comes from woody plants. Until we had systematic determinations of what deer eat, many thought a grass range was excellent deer range.

A pioneer investigation of food requirements of Colorado mule deer, carried on through 1938 to 1942, showed only one period of three months in which grass made up any appreciable part of their food. In spring, grass was 12.22% of total deer diet. Throughout the other 9 months grass was less than 1% of food taken. These data were based on stomach samples collected as 250 deer were range slaughtered for scientific examinations. Shrubby plants, leaves, buds, twigs, bark, and fruit supplied most of the deer food. And surprisingly, two kinds of evergreens, pinon pine and the western junipers, supplied approximately 35% of the winter diet of the mulies.

California found that in January deer ranging in the far northeastern corner of the state included evergreens as 26.4% by bulk of their diet. Even more significant was finding evergreen browse in 89% of the stomachs examined. Almost surely growing tips of young trees and branches were the portion of the evergreens browsed. The future of any young tree can be blighted, a whole generation of forest can be "eaten out" if only the tender, growing tips of the young trees are nipped off.

TOO MANY DEER HAMPER REFORESTATION

The problem of deer feeding on woody plants is not peculiar to western game ranges. At least *forty-seven studies* of feeding habits of white tail deer have been made in northern states, Maine through New York and Pennsylvania to Minnesota. White tails seem to prefer white cedar for food, with hemlock, the maples, and then the sumacs acceptable in that order. They feed, however, on the tips and buds of most of the better hardwoods and seem to particularly relish the tips of young planted fir, red pine, and white pine. Heavy deer populations make it impossible to successfully reforest many areas.

In the southern states, 11 studies supplied less positive definition of deer food requirements. In that region deer ate young growth of hardwood forest trees—maples, gums, and their associates. Curiously

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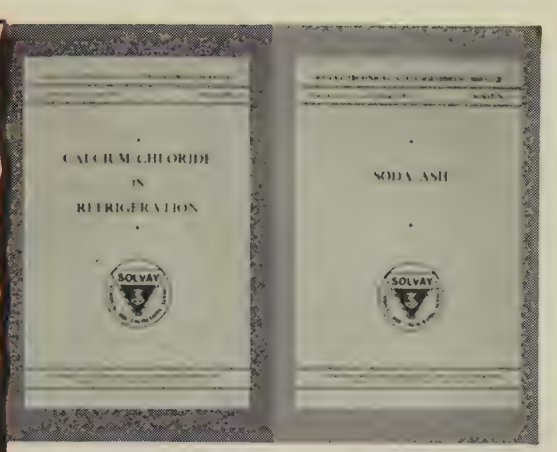

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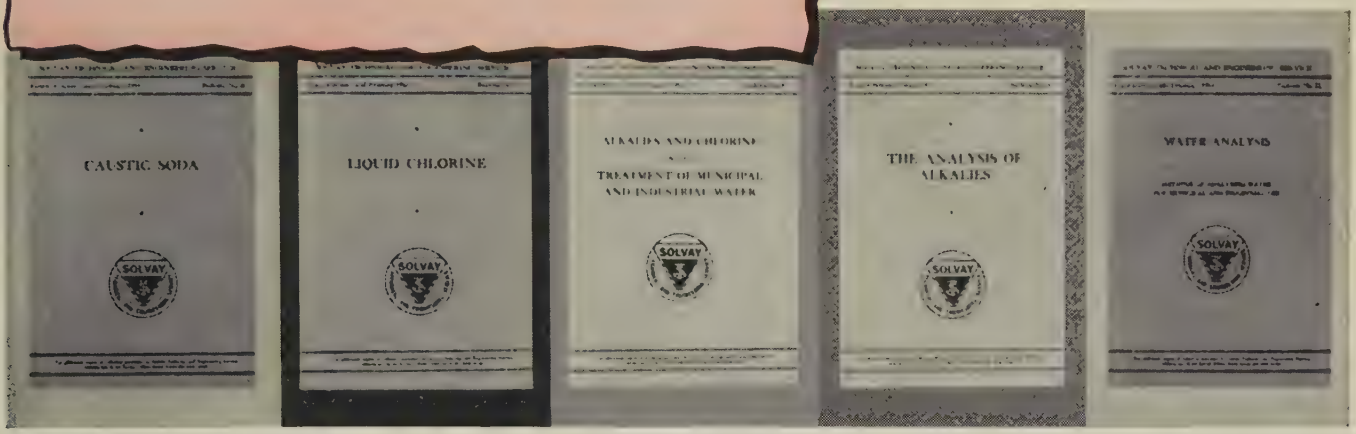

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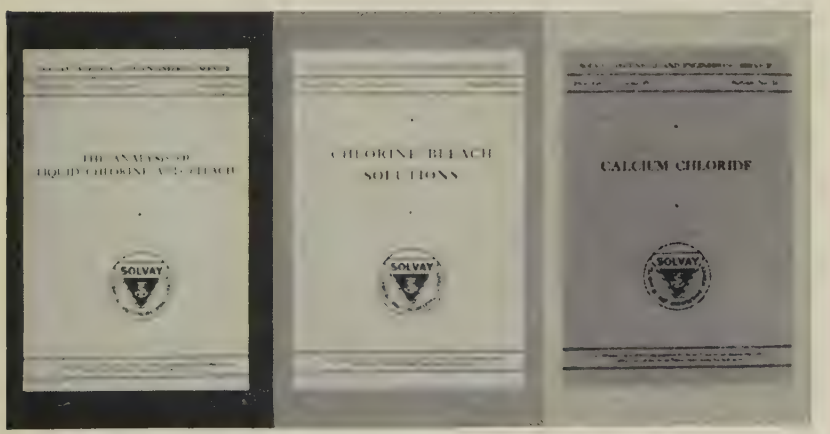

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The DRAPER Felt
DRAPER BROTHERS COMPANY
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in Texas and North Carolina, deer fed comparatively heavily on grass.

As early as 1931, investigators in Pennsylvania placed four yearling deer in a 5-acre test plot of typical mixed forest type. Between spring and September 10 these deer had eaten out all leafy growth and were beginning to chew on twigs and branches, though a goodly supply of grass was available. When a deer herd begins to overuse its range, damage in massive chunks is just ahead for both the carrying capacity of the range and the herd, or herds, depending on the food it supplies. Just how much damage may build up is well illustrated by the "peak" in Wisconsin deer populations that arrived about 1948.

A statewide check was made to determine how seriously the overabundant deer were hurting the young forests. The report came back—browsing deer were destroying young trees at a rate *one hundred times the loss that season caused by forest fires.* At that time forestry officials were expanding Wisconsin's state nurseries to grow 40 to 60 million seedling trees per year for planting on denuded land. That year deer were destroying *an estimated 600 million trees by browsing!*

Of course beavers destroy trees by cutting them down for food, flooding tree-growing sites, and building beaver houses. In many locations even greater damage to the forest is caused by the ponds drowning out tree roots.

Moose and elk take food from woody plants much as do deer. And if there is any question about bear doing damage to a forest, recently I inspected one 35-ft. fir tree in Grays Harbor County, Wash., that had at least half its trunk stripped to the solid wood. Within view were many other trees of like size that bears had thoroughly stripped of bark. Bears like sugar and it is believed this is the reason they tear off sweetish inner bark of younger conifers.

In every part of the country, with no regard for land ownership, some of our more important wildlife species do damage, particularly to future forests. Recently the American Forest Products Industries, Inc., made nationwide inquiries on wildlife damage on industrially-owned woodlands.

Altogether 46,263,852 acres were surveyed. Deer damage was reported by owners of 20,046,017 acres and was rated serous by the owners of 12,724,980 acres. Bear damage almost entirely in the West, was reported by the owners of 8,911,289 acres with serious damage on ownerships of 5,859,099 acres.

Across the nation, deer are principally browse eaters. We now should have a better understanding of what we mean by forest "edge." Specifically, in the case of the deer, it is browse, mostly bushes, twigs of young trees, and weedy growth with occasional use of grass.

Typical "edge" is found where forest surrounds a meadow. From the margin of the grassy open space, shrubs and vines build up through stages of tree-like shrubs, shrub-like trees, and the low-crowned trees, toward tall timber.

But "edge" used in reference to wildlife environment may spread over thousands of acres in continuous plant-type associations. It exists in the more open stands of some of the pine species, where an understory of shrubs and vines grows in semishade and where one or several old trees are selectively cut and young growth takes hold.

Most certainly, under modern methods of timber harvest, there always will be acres of "edge." It is the recurring creation of "edge" by maintaining forests on a sustained yield basis which calls for recurrent harvest of older trees that is the greatest promise, perhaps assurance, that in the future there will be habitat that can supply our most valuable wildlife with suitable homes.

HUNTING IS HARVESTING

The case of the Kaibab deer became the classic example of what happens to a deer herd when it begins to erupt. There probably were times and places where this happened before Michigan had its 1880–1900 build-up and die-off, and before the Kaibab rise and fall. The Kaibab became famous because of the big squabbling over who should order what in the way of hunting to harvest and make use of the surplus animals while the deer died by thousands.

The complete eruption and collapse pattern has, occurred in a number of places—including the squabbling over the basic issue of reducing the number of breeding does to stop the snow-balling increase in the herd.

Between 1910 and 1928, beginning with an estimated 1000 deer, plus large refuges, plus an iron-clad buck law, Pennsylvania's deer population exploded to in excess of 1,000,000 head—then hit the skids.

Michigan in 1937 again had a deer crisis. An esti-

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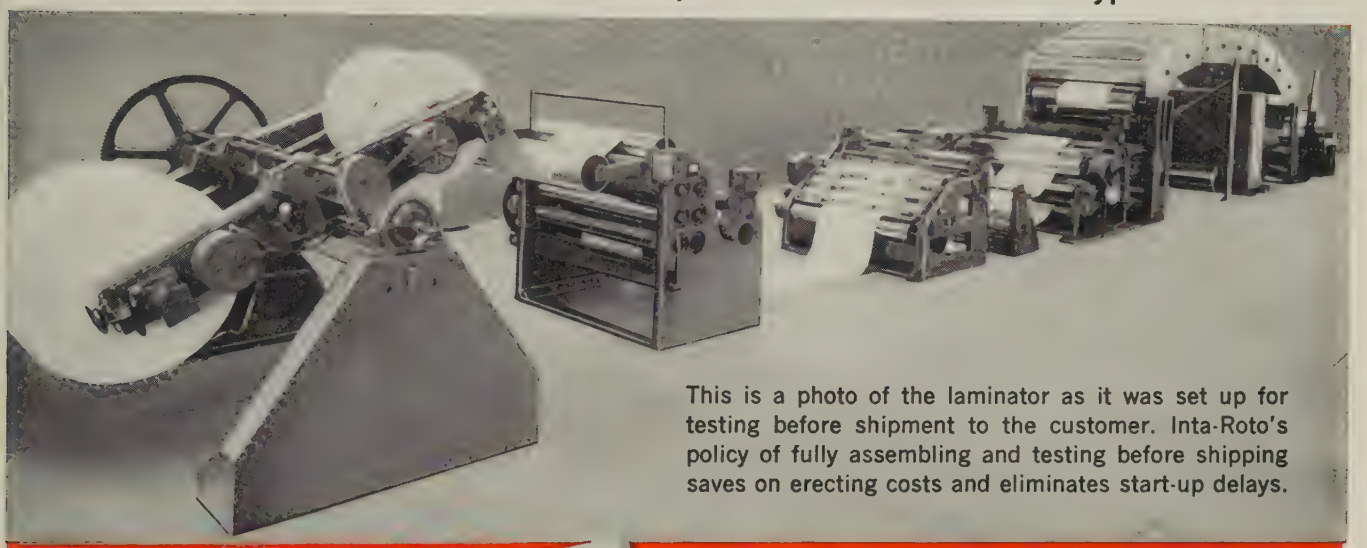
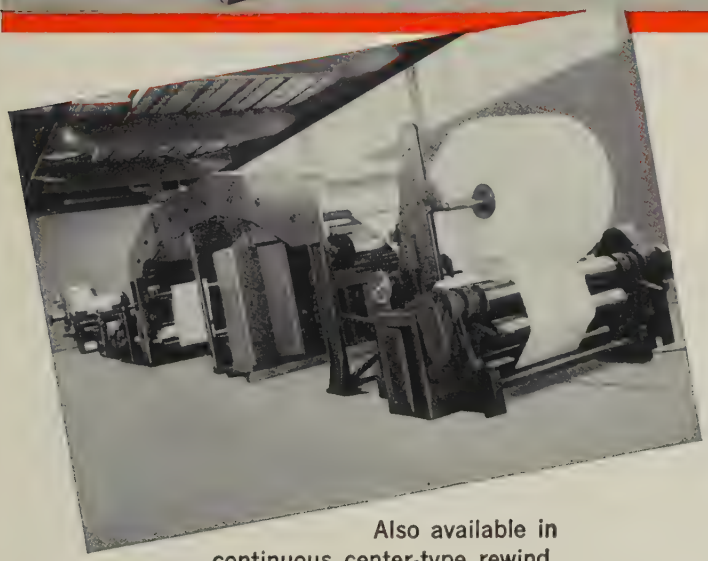


This is a photo of the laminator as it was set up for testing before shipment to the customer. Inta-Roto's policy of fully assembling and testing before shipping saves on erecting costs and eliminates start-up delays.



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mated 1,172,975 head roamed range which could properly take care of about 450,000. Inadequate liberalized hunting only partially blocked an all-out die-off.

In Colorado, where in the late 1930's and early 1940's ground alfalfa and stock food pellets were being fed to deer in an attempt to tide animals through the winter crisis time, nearly half the fawns died on one canyon feeding ground. Comparable losses occurred in other feed grounds, and after snow melted, game wardens working in one limited locality stacked up nearly 6000 carcasses of dead deer out of sight from the highway, threw oil on them, and lit the pyre. The range was capable of carrying a herd of 12,000 head on a sustained basis, yet a census showed 23,400 head of deer were on it, and the local people fought furiously against shooting does to check the population eruption.

A like pattern showed up about the same time on the Dixie National Forest in Utah. The deer herd built to a dangerous 50,000 head on range capable of supporting 35,000, and when officials tried to control the herd by reducing the does on it, sportsmen and politcos whooped protest. Men bought the special doe licenses, and while news cameras clicked, burned the licenses or tore them to pieces. It made news; it made no sense. After cooling off, Utah sportsmen saw the rightness of controlling herds by hunting harvest. They have checked back the herd several times and found it resurgent at an even greater increase rate (with more hunting as a result) each time the reduction has been made.

The list could be extended by many times if all comparable problems of population build-up among deer were listed. These are typical examples—the beginning of snow-balling in population, the faster-and-faster increase, the efforts of the officials who know what impends to get reduction of the productive does, the outcry against any killing of the "sacred doe," and then either the big die-off or some relief, some adjustment though late, as sportsmen finally are convinced harvest is better than mass starvation of the entire herd unit.

Until systematic records were kept, not much information on how fast deer might increase was available. We know now that potentially a healthy deer herd might *more than double in size in one season.* Here's why it *might* do so.

Between 1938 and 1943 some 250 range-slaughtered Colorado mule deer does were examined after the rut for evidence of pregnancy. Right across the board, including yearlings and "dry" does, 166 recognizable embryos were tallied for every 100 does.

Applying this to a deer herd in which there is one buck to each five does, the 100-doe herd, plus 20 bucks, would add up to 120. Add, now, the 166 fawns. The total would be a possible 286 animals. Carry it to the second season; if the ratio holds at one buck to five does, the 100-doe herd would be 183, for half of the fawns would be females. Bucks on at 1-to-5 ratio would number 36. The total breeding herd would would stand at 219. The theoretical maximum fawn crop would approximate 200 head—and half of them would be females.

California found in comparable studies that 100 does carried 173 embryos in one herd, 171 in another herd investigated, with a high of 175 in another part of the state. The lowest ratio found was 127 per 100 does.

In normal habitat deer never reach this maximum theoretical reproduction. But the approximate average increase is enough to shake one into realizing the imperative need to watch and control against eruption of population.

In Michigan, 1928, four does, two bucks were placed in a big enclosure, large enough to approximate natural conditions. Five years later that sextette had boomed up to 160 head.

Another case: 11 years after one buck, three does, were released in 1933 on the Horseshoe Lake National Wildlife Refuge in Illinois, the herd had grown to 250 head.

The wildlife research men figure an annual net increase of about 40% can be expected, and hunters may safely take about 25% each year of both sexes.

MOST DOES PRODUCE TWIN FAWNS EACH YEAR

Two significant sets of facts should be added to the deer data. First, though it was believed that doe fawns did not breed until they were well into their second year, it is now known certainly they may breed in their first year. The average life span of the doe is 8 to 12 years, and they are capable of producing twin fawns each year of their lives. It has been established that one buck can service as many as 20 does, but the optimum buck-doe ratio is one buck to five does.

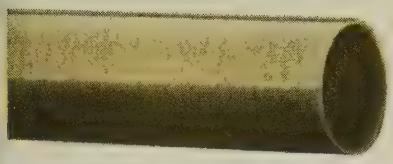
Devoted sportsmen once fought militantly to protect does; what they did then performed a great service in re-establishing our deer herds. They can do as great a service today by insisting on and lending a hand in harvesting excess she-stock before herd or flock populations start snowballing toward a disastrous overstocking of a game area. This is nobody's opinion as to what must be done to curb population explosions. It is common sense application of facts we now know about wildlife.

The other set of facts double-emphasizes the common sense of keeping livestock, though wild, in balance with its available food.

The *percentage* of fawns born decreases terrifically in the herd living on a beat-out range. The *survival* of the fawns falls off as drastically. The weights and condition of individual animals suffer extreme deterioration. In one herd where white tail bucks had been averaging 150 lb. hog-dressed weight, the weight dropped to 115 lb.

The rock-bottom truth is that a smaller breeding herd maintained on a top-condition range may produce a greater annual *crop* of vension, numbers and pounds, than a greater population with its winter-crisis loss due to malnutrition, disease, and parasite epidemics and common, outright starvation.

These are representative facts of wildlife management that sportsmen, foresters, and game and fish officials now have with which they may work out full, well-balanced programs for the wood-growing Tree Farm and the wildlife that inevitably utilizes that managed forest as its home. Deer has been used here as the species to illustrate essential conditions that may be present on most timberlands. Deer was used as the illustrative species also because it is so universally established as our main type of big game, and because



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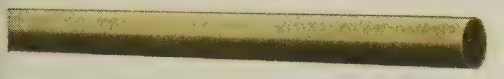

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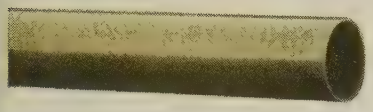

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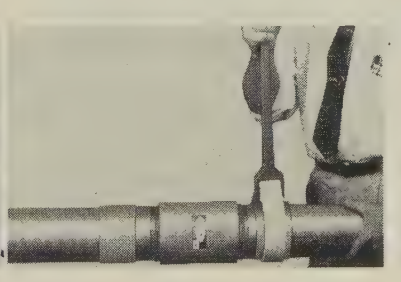

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The resistance of BONDSTRAND pipe to chemical solutions and wet environments make it well suited to pulp and paper operations, including the transportation of pulp, stock, water, wastes and dyes. Tests are recommended for liquor applications.

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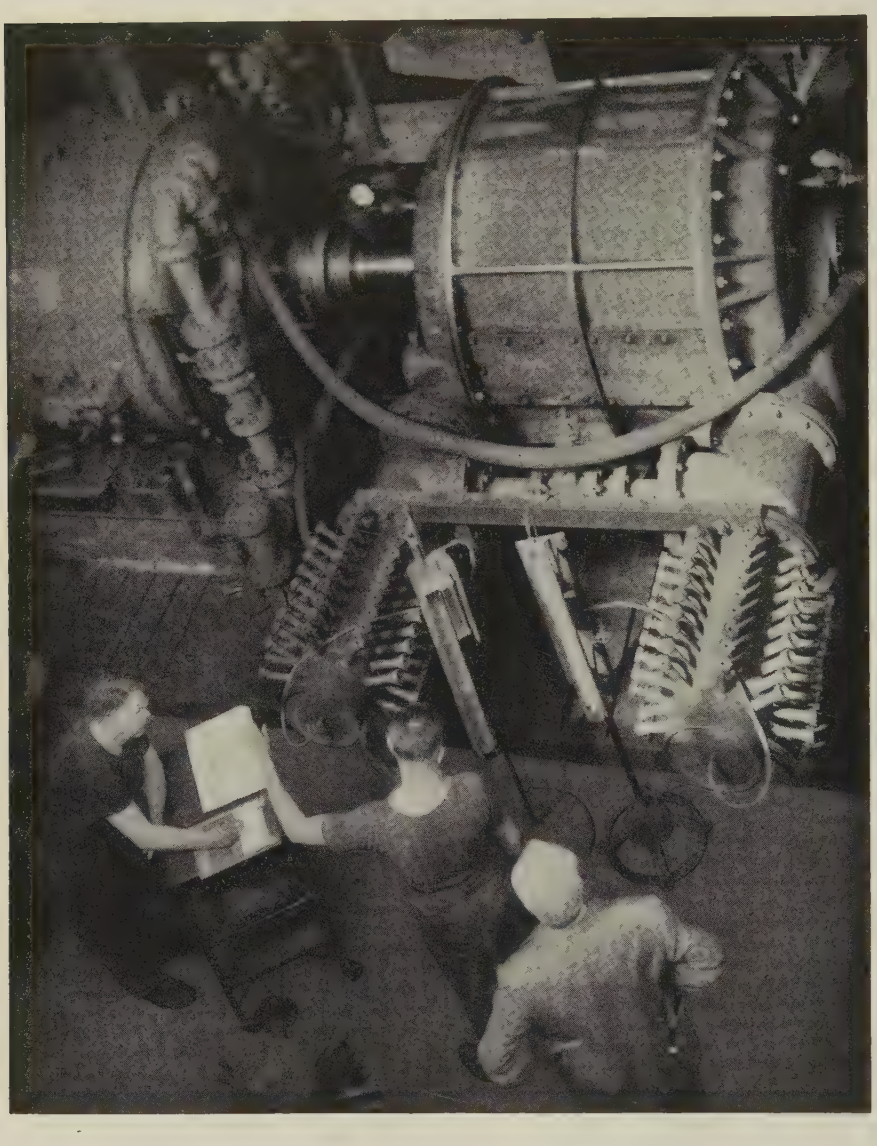
Dept. YI • 4809 Firestone Boulevard • South Gate, California

921 Pitner Avenue, Evanston, Illinois
360 Carnegie Avenue, Kenilworth, New Jersey
2404 Dennis Street, Jacksonville, Florida
6530 Supply Row, Houston, Texas

A Subsidiary of American Pipe & Construction Company

Rated performance of every Nash Vacuum Pump is assured by this precise laboratory test



Rated capacities of Nash Vacuum Pumps are not theoretical. Every Nash Pump is tested individually. Air capacity is determined by delivery thru accurately machined and calibrated orifices. Related vacuum is measured by precise mercury column, and horse power is recorded electro-dynamically. Records of these tests are retained by us, and certified copies are available to Nash Pump owners.

That is one of the reasons why Nash Vacuum Pumps are installed in over a thousand leading Paper Mills. An engineer from Nash will be glad to survey your mill, and make recommendations, entirely without obligation to you.

NASH ENGINEERING COMPANY

440 WILSON ROAD, SO. NORWALK, CONN.

we have such positive evidence that failure to harvest surpluses as a crop can cause great damage to both game and its habitat.

In varying degrees, the principles of wildlife management are similar in most localities with reference to most species.

Permit me now to be direct and personal. I've tried to supply you, whether you're forester, landowner, game official, or sportsman, with enough bed-rock facts on which all can develop teamwork in insuring good management plans for the twin crops of trees and game. There's a potential, in the Tree Farm movement and all forest management like it, to materially increase not only timber supplies, but game resources.

The opportunity's too good to pass by; grab hold, brother outdoorsmen, and let's get our part of the job going!

13th

Alkaline

Pulping

Conference

of the

Technical Association

of the

Pulp and Paper

Industry

Hotel Robert Meyer

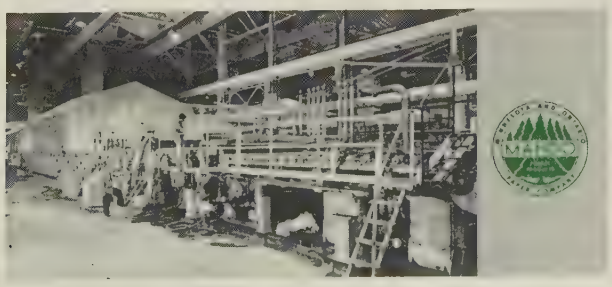
Jacksonville, Fla.

Nov. 4–6, 1959

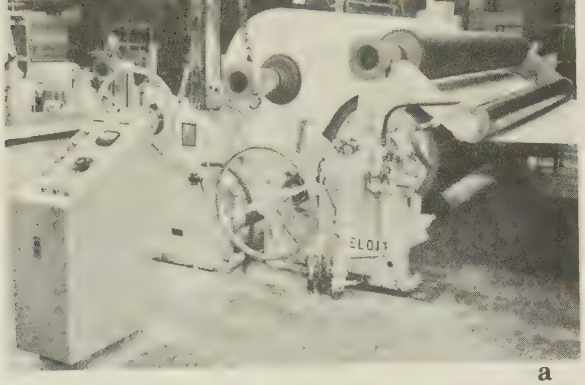
S. R. CHRISTENSEN

Minnesota & Ontario Paper Company Number 2 Machine, International Falls, Minnesota

Minnesota & Ontario Paper Company



MANDO'S new 158″ publication grade machine marks the completion of an extensive modernization program at International Falls. For details of this versatile, new machine, especially designed for a wide range of publication grades, please turn the page.



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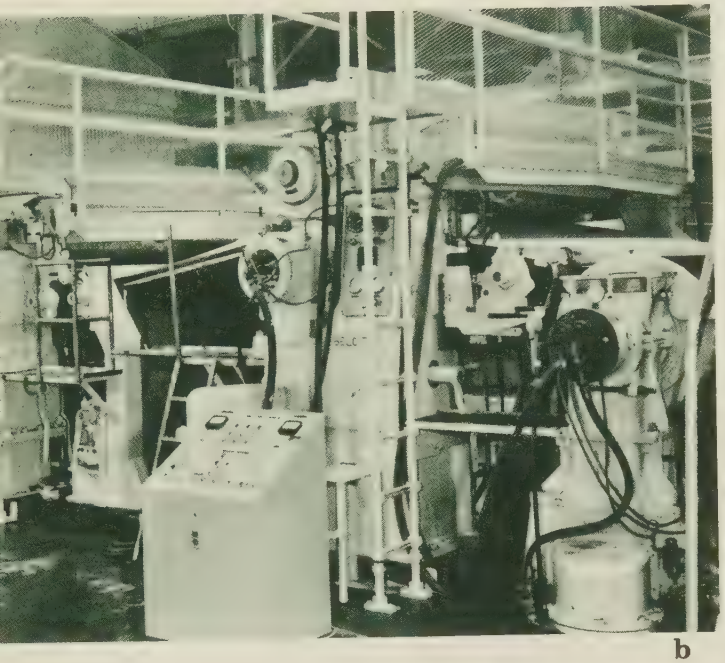

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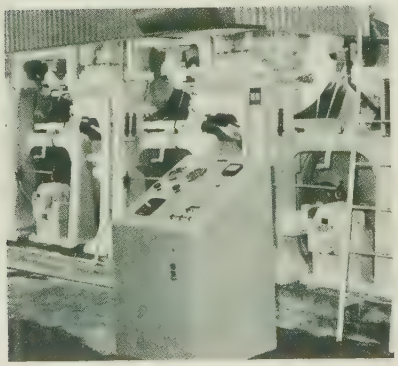
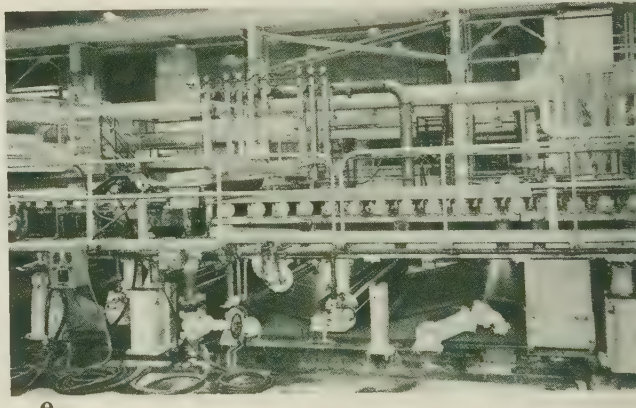


. . . some features of Mando's new Beloit 158″ publication grade machine pictured on the preceding page

The start-up of the new No. 2, 158″ Beloit machine now brings MANDO'S production at International Falls to 270 tons per day. This versatile machine, for publication grades, is designed for operating speeds in the 1500 fpm range. Pictured on this page are some of the machine's features: (**a**) transfer type reel; (**b**) straight-through suction presses, suction couch, and plain smoothing press; (**c**) Beloit horizontal size press—located between second and third dryer sections; (**d**) dryer section—enclosed gear-type drive, antifriction bearings serviced by Beloit roll filter lubrication; (**e**) 158″ Beloit removable, shakable Fourdrinier; (**f**) oscillating unwind stand and water-cooled brake.



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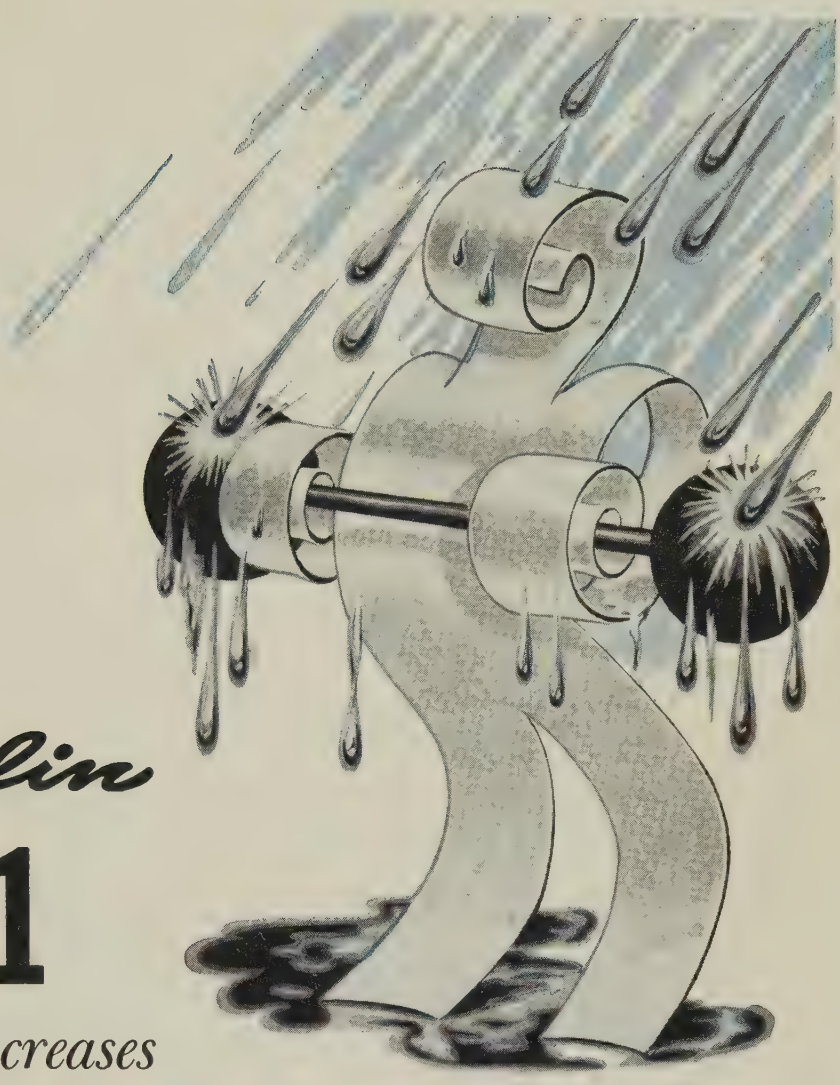
BELOIT

PAPER MACHINERY

your partner in papermaking



NEW!

Catalin RESIN 301

substantially increases

WET STRENGTH

without increasing cost

Because it is more efficient, Catalin Resin 301 . . . a modified, cationic urea-formaldehyde resin . . . can substantially upgrade wet strength with the same percentage of resin solids previously used . . . or cut resin costs by maintaining present wet strength properties with less resin solids. You gain in other ways too . . . Catalin Resin 301 is lighter in color . . . and reacts exceptionally well in the presence of dyes. Dry tensile, mullen and folding endurance is also improved.

Usable in a wide variety of bleached and unbleached pulps, Catalin Resin 301 develops about 50% of its final wet strength as it leaves the machine . . . full cure is reached within 2-3 weeks. Since wet strength is developed slowly, broke can be easily reclaimed.

Samples, specification sheets, laboratory data and technical assistance are yours for the asking. Catalin welcomes your inquiry.

Catalin

Catalin Corporation of America
One Park Avenue, New York 16, N. Y.

PLANTS:
Fords, New Jersey • Calumet City, Illinois
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How B&W JOB-MATCHED TUBES provide long service life in stainless steel

When you specify B&W Stainless Heat-Exchanger Tubes—either seamless or welded—you can count on:

- ... a complete range of stainless grades to meet any set of service conditions
- ... a wide variety of diameter and wall thickness combinations for all types of operating requirements
- ... fully annealed tubes for maximum resistance to corrosion

These are just a few of the reasons it pays to specify B&W Job-Matched Stainless Steel Heat-Exchanger Tubes. Call your local B&W District Sales Specialist, or write for Bulletin TB-329 for full information. The Babcock & Wilcox Company, Tubular Products Division, Beaver Falls, Pennsylvania.

CHEMICAL SHOW • Booth 1002
Coliseum • New York
November 30—December 4

B&W

THE BABCOCK & WILCOX COMPANY

TUBULAR PRODUCTS DIVISION

TA-9019-S3

Seamless and welded tubular products, solid extrusions, seamless welding fittings and forged steel flanges—in carbon, alloy and stainless steels and special metals

Pump enough water in two minutes to make two thousand lbs. of paper!

To make a ton of paper, you need approximately 12,500 gallons of water.

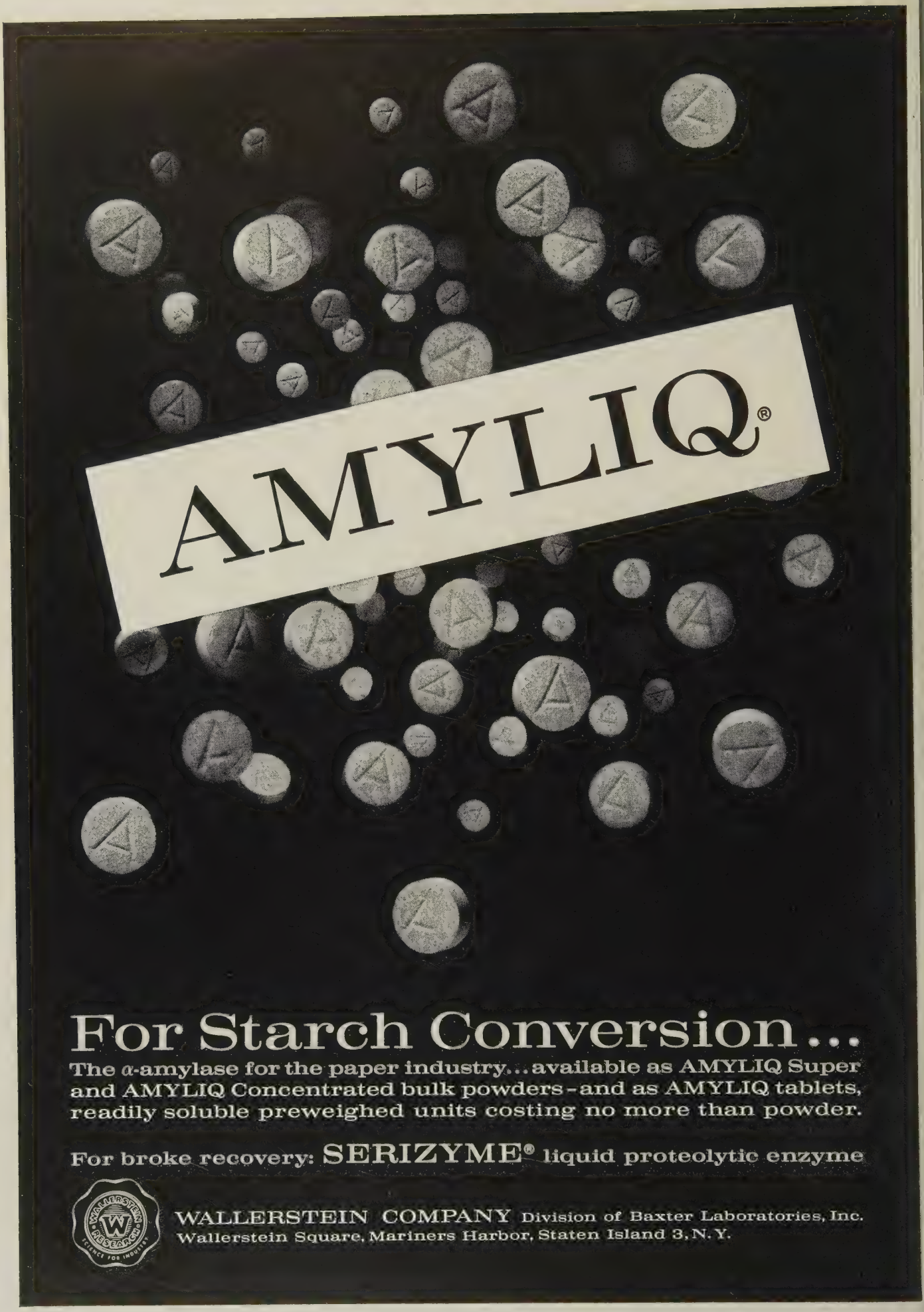
It would take just *two minutes* for a twelve-inch Goulds Fig. 3405 to supply it. This medium-size pump provides giant-size capacity: 6400 gallons per minute!

And to keep water flowing, you stock only a minimum of spare parts. Just three groups of parts fit thirty-three sizes of the 3405. All parts are interchangeable except casings, casing covers and impellers!

That means that for every three to five pumps of a given group size, you stock just one shaft, one set of bearings, sleeves, mechanical seals or packing. You can cut inventories as much as ninety-one percent, yet be fully covered for emergencies.

Heads go up to 425 feet. For more data, write to Goulds Pumps, Inc., Dept. TA-89, Seneca Falls, N. Y., for Bulletin 721.6.

GOULDS GP PUMPS



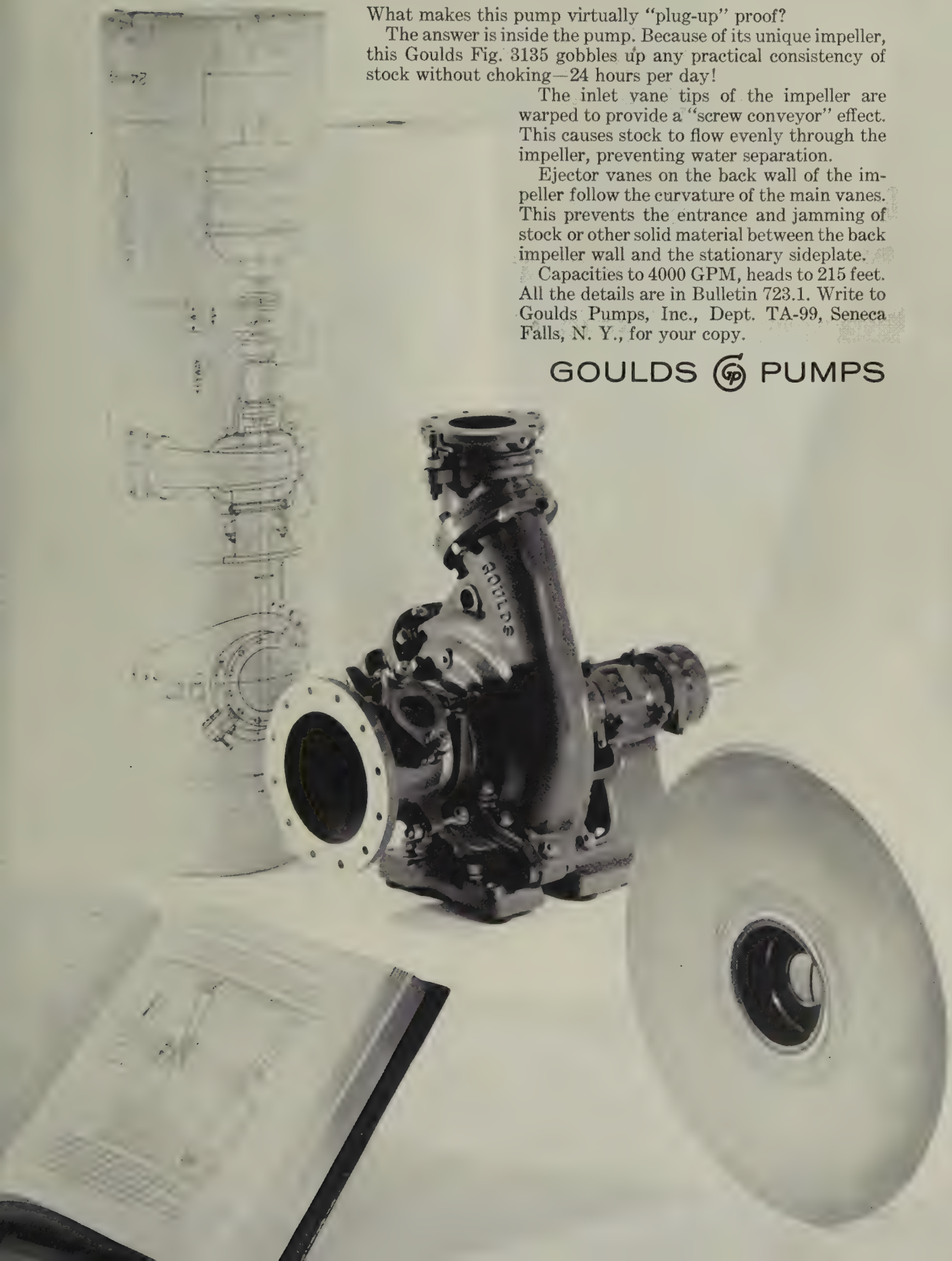
For Starch Conversion...

The α-amylase for the paper industry... available as AMYLIQ Super and AMYLIQ Concentrated bulk powders - and as AMYLIQ tablets, readily soluble preweighed units costing no more than powder.

For broke recovery: SERIZYME® liquid proteolytic enzyme

WALLERSTEIN COMPANY Division of Baxter Laboratories, Inc.
Wallerstein Square, Mariners Harbor, Staten Island 3, N. Y.

The pump that refuses to choke on paper stock!



What makes this pump virtually "plug-up" proof?

The answer is inside the pump. Because of its unique impeller, this Goulds Fig. 3135 gobbles up any practical consistency of stock without choking—24 hours per day!

The inlet vane tips of the impeller are warped to provide a "screw conveyor" effect. This causes stock to flow evenly through the impeller, preventing water separation.

Ejector vanes on the back wall of the impeller follow the curvature of the main vanes. This prevents the entrance and jamming of stock or other solid material between the back impeller wall and the stationary sideplate.

Capacities to 4000 GPM, heads to 215 feet. All the details are in Bulletin 723.1. Write to Goulds Pumps, Inc., Dept. TA-99, Seneca Falls, N. Y., for your copy.

GOULDS PUMPS

Photo of Tower of Parliament and Big Ben Clock by Ewing Galloway

HAM FELTZ says:

"Reminds me of HAMILTON Felts . . . they run a long time, too!"

"Since 1858—the year before "Big Ben"—the famous clock was installed in London's House of Parliament —papermakers have found that Hamilton Felts give longest high quality performance between changeovers.

They help you reduce the high cost of downtime by keeping your machines running at top speed —longer . . . turning out tonnage with less broke and imperfections.

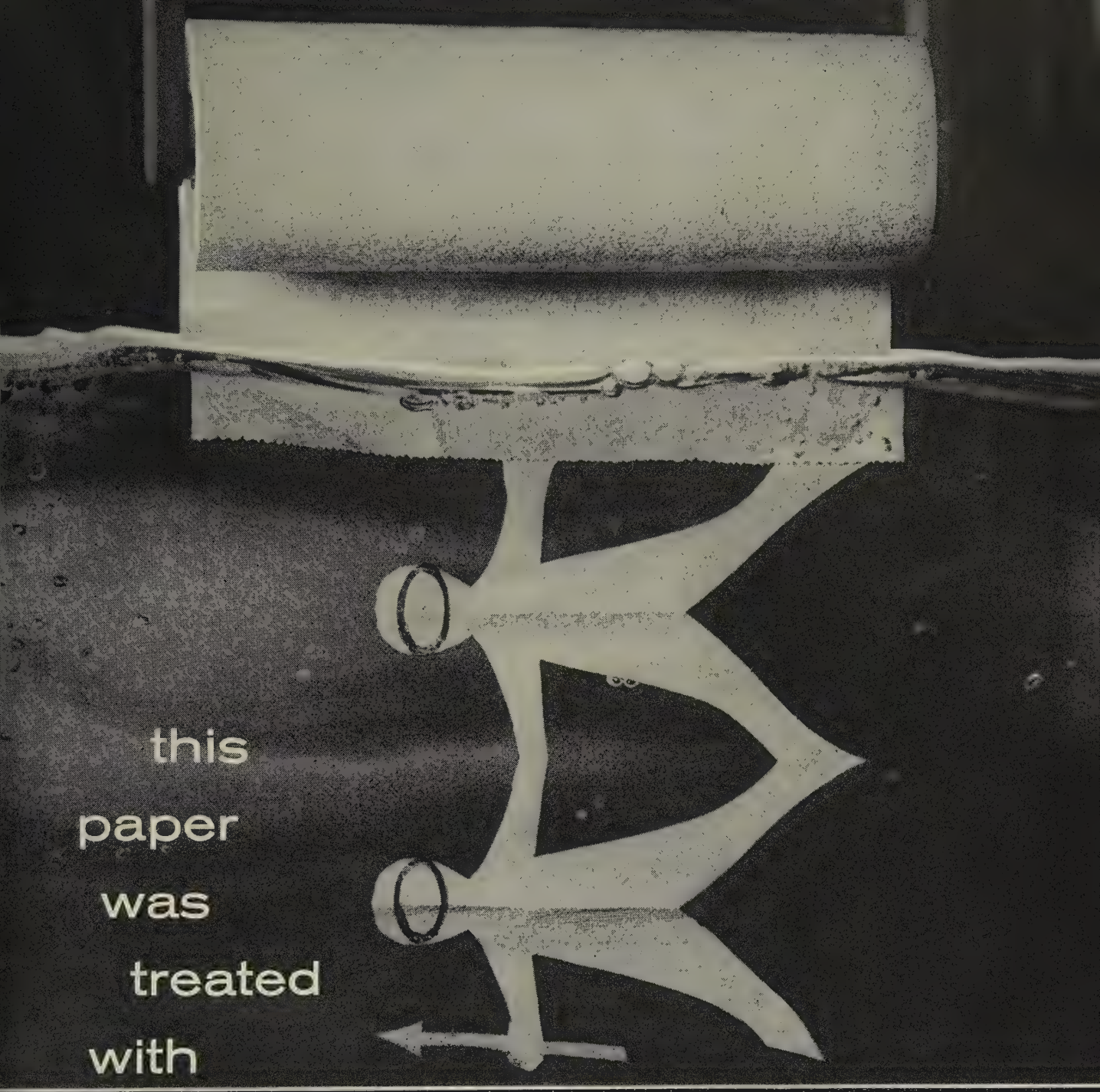
When it's time for a new felt on one of your machines, why not start using us as one source of your felt supply? Write, wire or telephone—today!

YOU CAN'T BEAT

Hamilton

FELTS

SHULER & BENNINGHOFEN, HAMILTON, OHIO



SCRIPTITE* 40

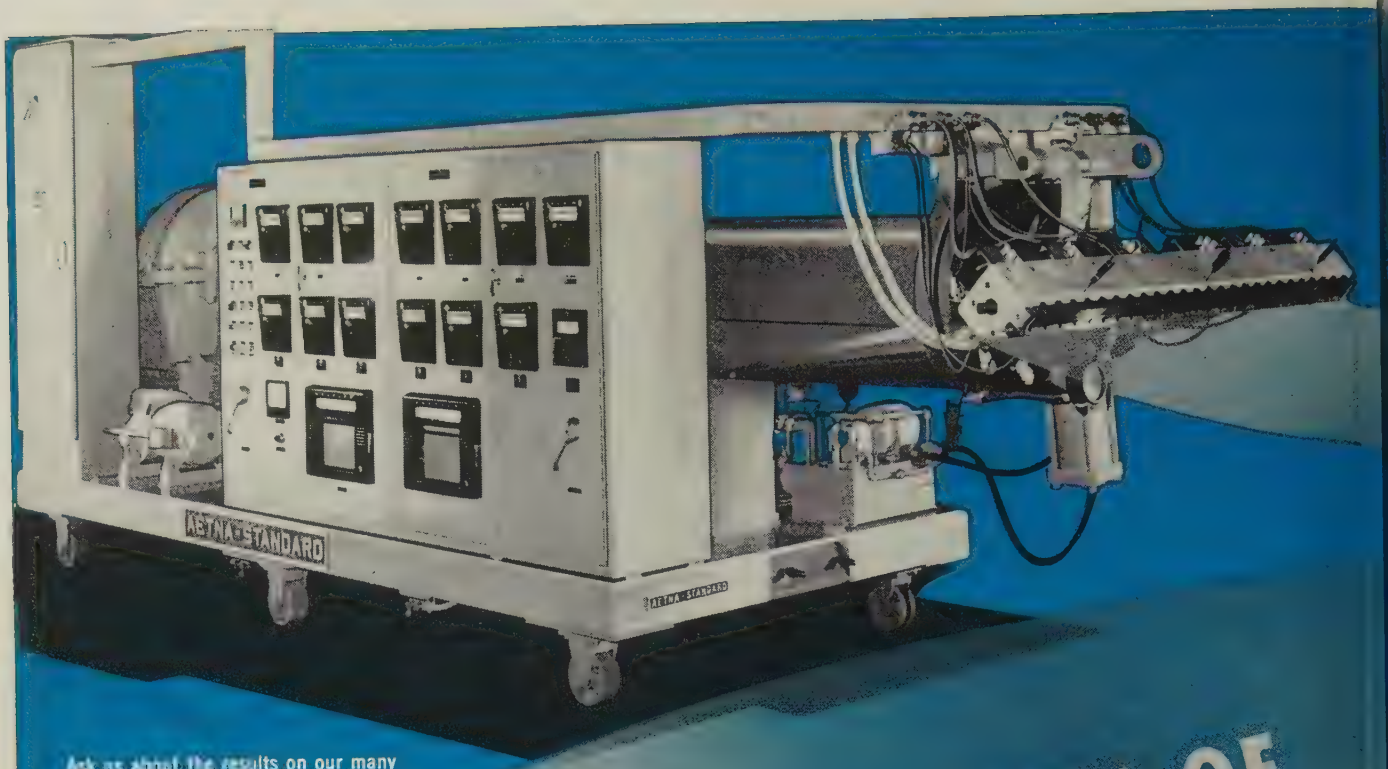
Scriptite 40, a new concept in urea-formaldehyde paper resins, imparts high off-machine wet strength with less resin add-on. Made more cationic, this water-soluble resin is strongly attracted to the cellulose, assuring greater application efficiency. Formulated for fast ure, Scriptite 40 also improves dry tensile strength, dry mullen, wet rub resistance, dimensional stability, and folding endurance. For samples and technical data, write: Monsanto Chemical Company, Plastics Division, Room 1154, Springfield 2, Mass.

Monsanto

*SCRIPTITE, LYTRON: Reg. U.S. Pat. Off.

The Monsanto line of paper resins also includes

SCRIPTITE 54 *outstanding water resistance, both wet and dry rub resistance.* **SCRIPTITE 55** *a low viscosity resin for improved water resistance, wet and dry rub resistance with easy handling.* **SCRIPTITE 52** *in combination with formaldehyde, gives water resistance to folding boxboard and jute liner.* **SCRIPTITE 50** *unsurpassed printability, improved surface characteristics on boxboard.* **SCRIPTITE 33** *melamine wet-strength resin.* **SCRIPTITE 45** *new thermosetting resin for stabilization of paper.* **LYTRON*** *water dispersal resin polymers for coatings.*



Ask us about the results on our many induction heated extruders in use.

HIGH SPEED PRODUCTION OF UNIFORM GAUGE OVERWRAP FILM now available using Aetna-Standard Induction Heated Extruders and Dies

INDUCTION HEATED DIES

Include all the features required for uniform gauge control: induction heating of the insulated body provides uniform temperature throughout the die; a unique design of leak-proof deckle equipment minimizes trim width; simple land adjustment, in combination with multi-zone temperature controls, minimizes run-in time.

500 F. P. M. and Up of Quality Product

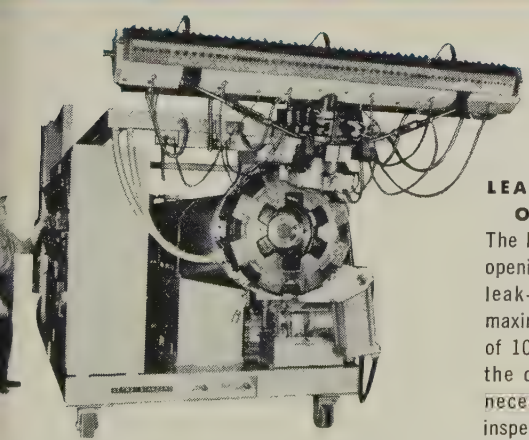
CONTROLLED PRESSURE ASSURES QUALITY... The controlled pressure plasticizing action of the screw can be maintained independently of die pressure or screen pack conditions.

HIGH SPEED SCREWS accurately designed for adiabatic operation assure the ultimate in output rates.

INDUCTION HEATING SYSTEMS, including automatic water-cooling equipment, provide "on the button" temperature control.

UNITIZED CONSTRUCTION . . . Just set it on the rails and connect the power supply.

COMPLETE INSTRUMENTATION . . . Recording of speed, melt temperatures, melt pressures, provides a complete record of performance.



LEAK-PROOF QUICK OPENING HEAD
The breech-lock-type, quick opening die adapter assures leak-proof sealing at the maximum design pressures of 10,000 p. s. i., plus all the quick opening features necessary for screen pack inspection.

HALE AND KULLGREN, INC. • AKRON, OHIO

SPECIALISTS IN MACHINES AND PROCESSES FOR RUBBER AND PLASTICS

AETNA STANDARD
DIVISION BLAW-KNOX COMPANY
PITTSBURGH, PENNSYLVANIA

WORLD WIDE PULPSTONE SERVICE!

Behind the world-wide acceptance of Norton pulpstones is a world-wide service organization of Norton pulpstone engineers — a group of men qualified by practical paper mill experience to study your mill and to make specific recommendations based on your type of wood, your kind of grinders and the type of pulp you want to produce.

No matter where your mill is located, there's a Norton pulpstone engineer within reach. He will be glad to specify the correct pulpstone to meet your particular requirements and to render technical service.

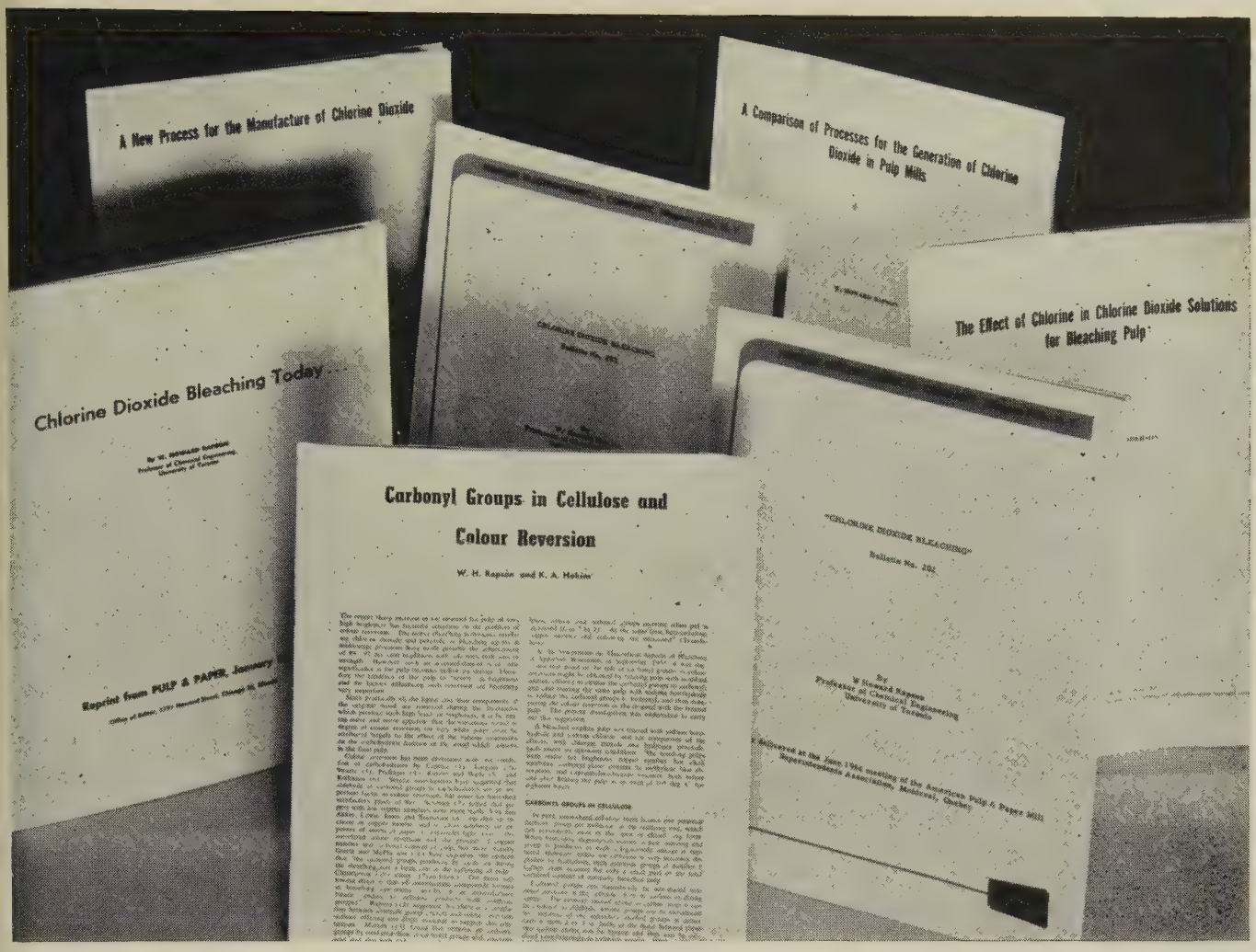
Hundreds of pulpstones are shipped each year from the Norton plant at Worcester and from Norton Company of Canada's plant at Hamilton, Ontario. Each has been carefully engineered to meet the special requirements of individual mills in all parts of the world.

Making better products . . . to make your products better

NORTON COMPANY, WORCESTER 6, MASS.
Norton Company of Canada Ltd., Hamilton, Ont.
EXPORT: Norton Behr-Manning Overseas Inc., Worcester 6, Mass.

NORTON PULPSTONES

Abrasives - Grinding Wheels - Grinding and Lapping Machines Refractories - Electrochemicals - Non-slip Floors | Norbide Products


A New Process for the Manufacture of Chlorine Dioxide
A Comparison of Processes for the Generation of Chlorine Dioxide in Pulp Mills
Chlorine Dioxide Bleaching Today
The Effect of Chlorine in Chlorine Dioxide Solutions for Bleaching Pulp
Carbonyl Groups in Cellulose and Colour Reversion
W. H. Rapson and K. A. Hobson
"CHLORINE DIOXIDE BLEACHING"
Bulletin No. 202
Reprint from PULP & PAPER, January

CHLORINE DIOXIDE ROUNDUP

Above you see a collection of the more significant papers and bulletins published on chlorine dioxide.

At right you see a coupon which you are invited to use to send for those pieces you find most interesting.

This roundup is typical of the information collected by the Hooker Technical Service Department on chemicals used in the pulp and paper industry.

Gathering and disseminating such information is an integral part of our service to you who use such Hooker chemicals as sodium chlorate, chlorine, and caustic soda.

CHECK ITEMS YOU WANT:

- ☐ Bul. 222 Materials of Construction for ClO_2 Manufacture and Bleaching
- ☐ Bul. 221 A new process for the Manufacture of ClO_2
- ☐ Bul. 220 Chlorine Dioxide Bleaching Today
- ☐ Bul. 219 Carbonyl Groups in Cellulose and Color Reversion
- ☐ Bul. 218 Effect of Chlorine on Chlorine Dioxide Solutions for Bleaching Pulp
- ☐ Bul. 205 Comparison of Processes for the Generation of ClO_2 in Pulp Mills
- ☐ Bul. 203 Chlorine Dioxide Bleaching

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HOOKER CHEMICAL CORPORATION

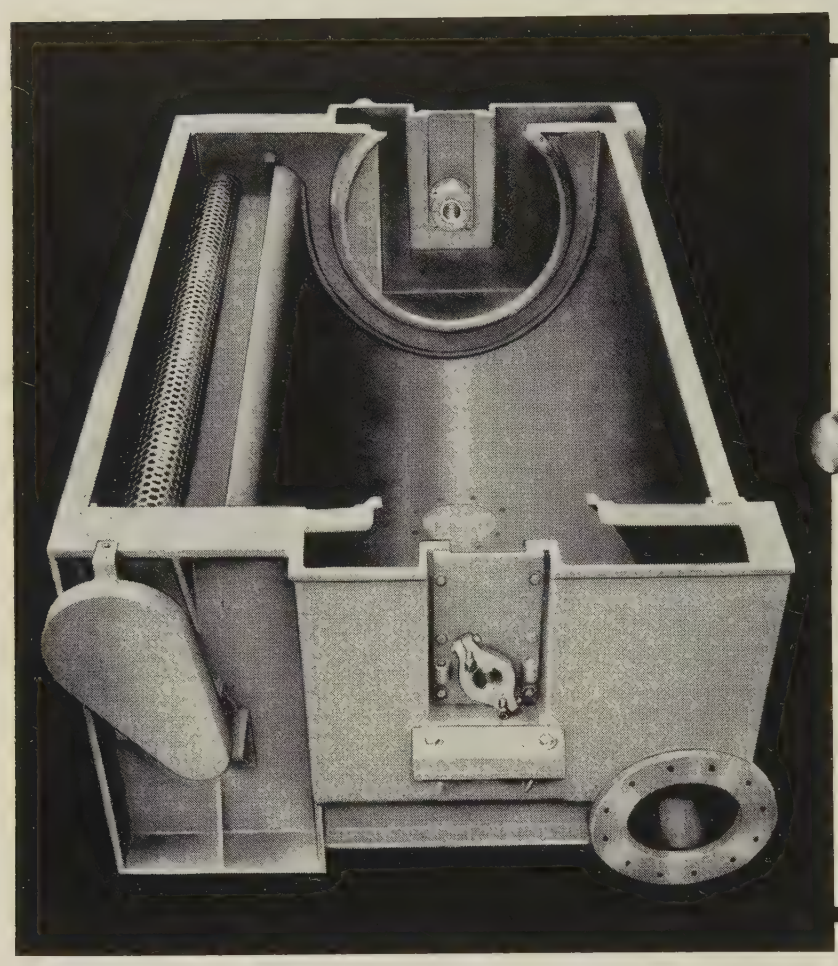
2109 FORTY-SEVENTH STREET, NIAGARA FALLS, N. Y.

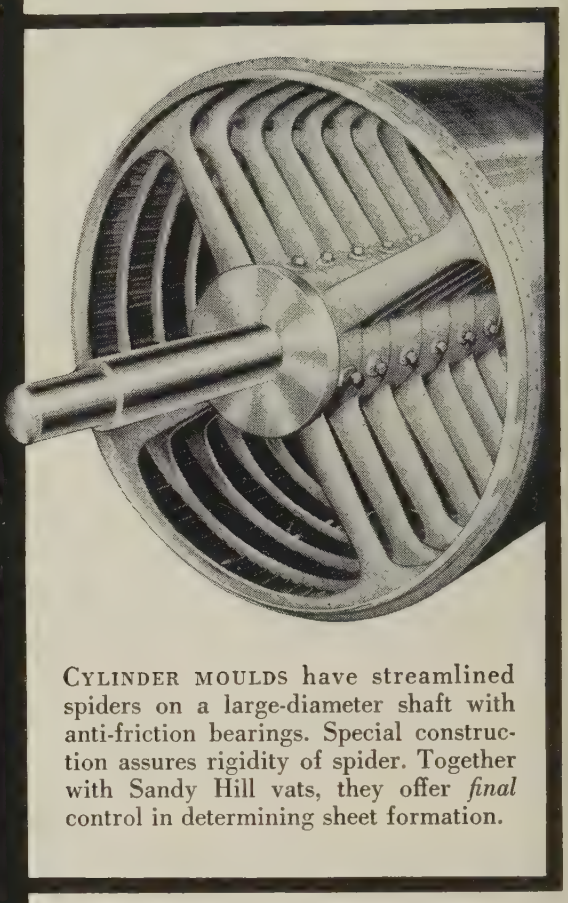
Sales Offices: Chicago Detroit Los Angeles New York Niagara Falls Philadelphia Tacoma Worcester, Mass.

In Canada: Hooker Chemicals Limited, North Vancouver, B. C.

HOOKER CHEMICALS PLASTICS

SANDY HILL CYLINDER MOULD VATS help you make better paperboard





CYLINDER MOULDS have streamlined spiders on a large-diameter shaft with anti-friction bearings. Special construction assures rigidity of spider. Together with Sandy Hill vats, they offer *final* control in determining sheet formation.

SANDY HILL VATS offer superior inflow and outflow capacity. Inflow distributor roll gives better fiber dispersion and uniformity of cross-machine caliper. Can be all stainless steel, cast iron ends with cypress circle and baffles, or copper-or-monel-lined cypress.

Illustration shows counterflow vat. Direct flow vat available.

Tell us your vat requirements in terms of your product and your mill's production aims. Then, let us put your experience and ours together and see about reaching those aims in the most economical way. This kind of cooperation has led to more profitable mill operation in every part of the country!

• ***Phone or write today, for full information.***

THE
SANDY HILL
IRON AND BRASS WORKS
HUDSON FALLS, N. Y.

Just what you'd expect from the leader!

Three new modified gum rosin sizes now in use across the country !

Trust the leader in the industry to come up with the first and the finest. Cyanamid's vast, diversified and expanding research program is constantly up-grading and improving all Cyanamid products. And our three, fine, new *modified gum* sizes have been developed out of this research. Look them over yourself. They offer new economies in shipping and storage (Cyanamid's 80% solids fortified size is an exclusive in the industry) . . . minimum foam characteristics for top size efficiency (you can make less go farther, or use the same amount for a more highly sized end product) . . . and better color, for highest paper brilliance.

NEW! **ACCOBRITE® ROSIN SIZE 4028**
—Unfortified modified gum rosin size . . . shipped as 80% solids. Has excellent color, low foam. Ideal for use in board . . . and much lower in cost than fortified size. One of the lightest-colored sizes available—and the most efficient!

NEW! **CYFOR® ROSIN SIZE 4016A**
—A top-grade, fully fortified, 70% solids, modified gum size. Its low foam means highest size efficiency . . . and its good color (and competitive price) make it an excellent choice for mills making book, bond, ledger and board.

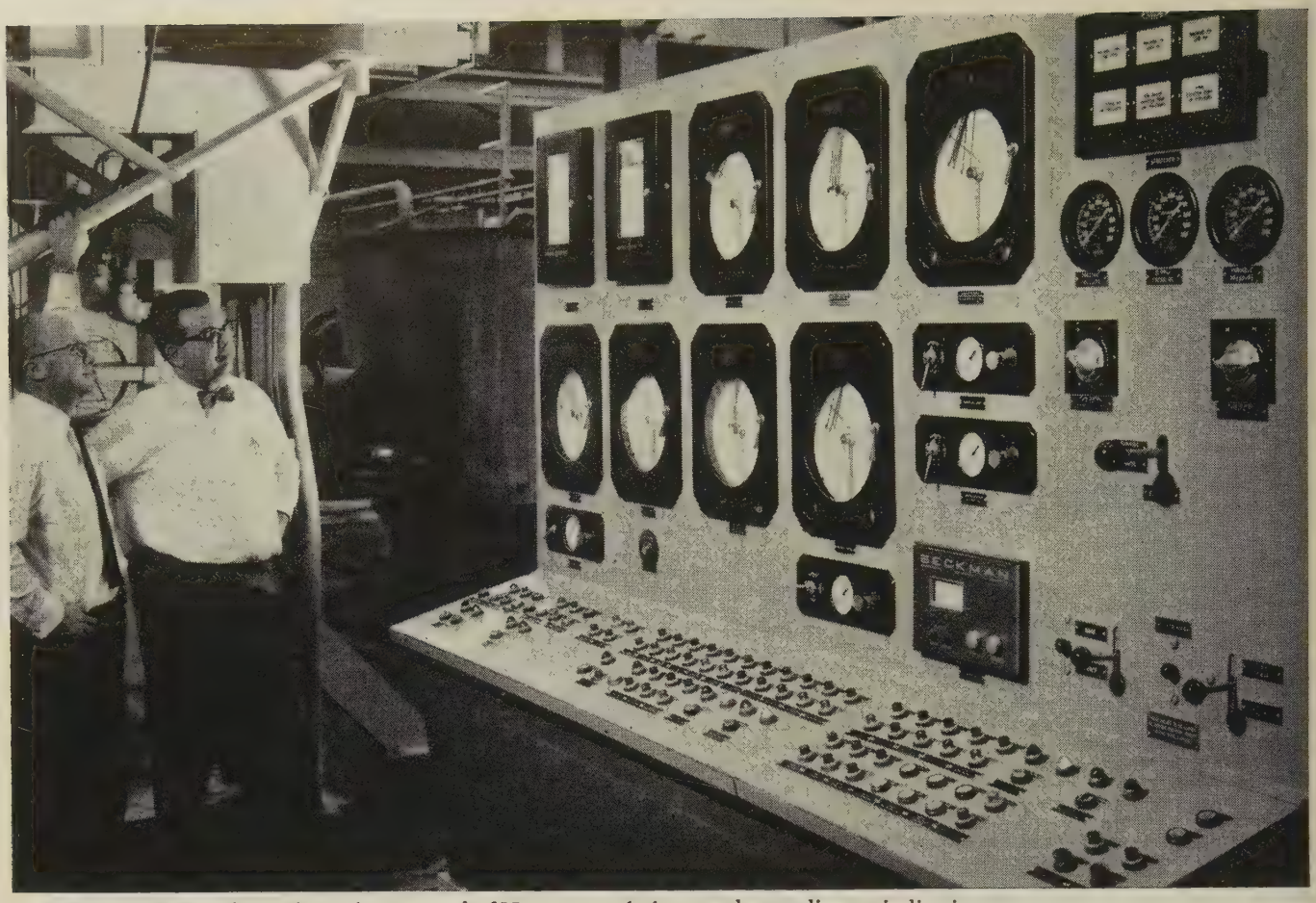
NEW! **CYFOR® ROSIN SIZE 4029**
—Save on shipping and storage with this exclusive, 80% solids, fortified size! Cyanamid is the only manufacturer making it. What's more, the General Electric Brightness Indicator shows this size to have extra-light color. It contains the same high-quality gum rosin used in 4028!

These three new sizes are just a small part of Cyanamid's great family of sizes. At Cyanamid you'll find more sizes, and more types of sizes, than anywhere else! Count on Cyanamid always for the best possible sizes—at the best possible price. It will pay you to call your Cyanamid Paper Chemicals representative to talk over your size needs. He's equipped to help you with the best knowledge in the industry!

CYANAMID

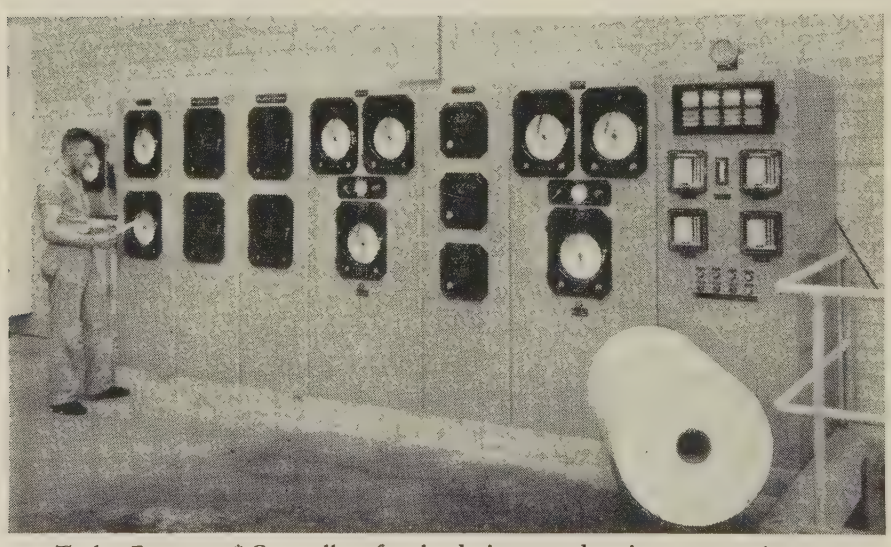
AMERICAN CYANAMID COMPANY
PAPER CHEMICALS DEPARTMENT
30 Rockefeller Plaza, New York 20, New York
In Canada: Cyanamid of Canada Limited, Montreal and Toronto

LARGEST SUPPLIER OF CHEMICALS TO THE PAPER INDUSTRY

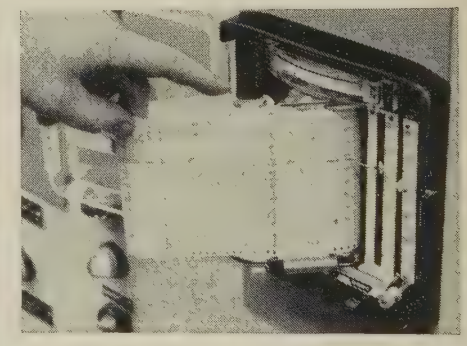


Control panel at wet end of No. 10, regulating—and recording or indicating—vacuum, pressure, pH, wire speed and level.

Whatever the Process...



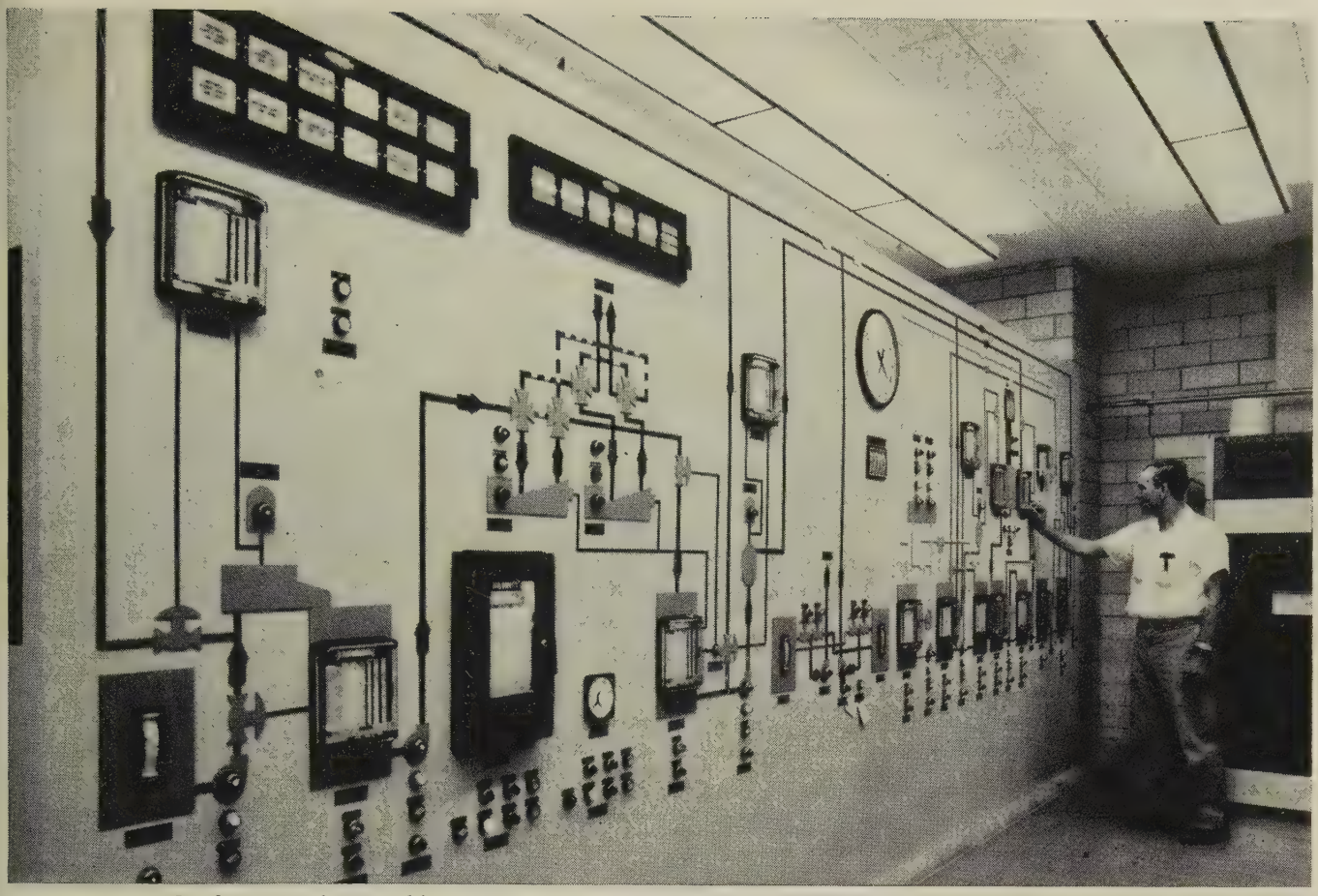
Taylor FULSCOPE* Controllers for the drainage and moisture control system supplied by Stamm Drying Controls, Inc.



Ecusta was the first paper mill to make wide usage of TRANSCOPE Recorder advantages: —the convenience of plug-in features for quick change; unconfused readability; servo-operated pens and process alarms. No other recorder puts so many features in so little panel space.

*Reg. U.S. Pat. Off.

Taylor Instruments



Stock preparation graphic control panel utilizes the many operating and maintenance advantages of the TRANSCOPE* Plug-in Recorder.

Ecusta relies on Taylor!

From baled pulp to finished paper, new No. 10 machine uses automatic controls

THE world's widest—and probably fastest—machine for lightweight paper was recently installed at Ecusta Paper Division of Olin Mathieson Chemical Corp., Pisgah Forest, N. C. The 182 inch wide No. 10 is probably the industry's closest approach yet to "Full automation".

Automatic controls monitor all stages of stock preparation, supervise the process flow and keep the specifications for the sheet constant. In the event of a break in the sheet, process flows are automatically reduced, production is cut off behind the break, and alarms are sounded.

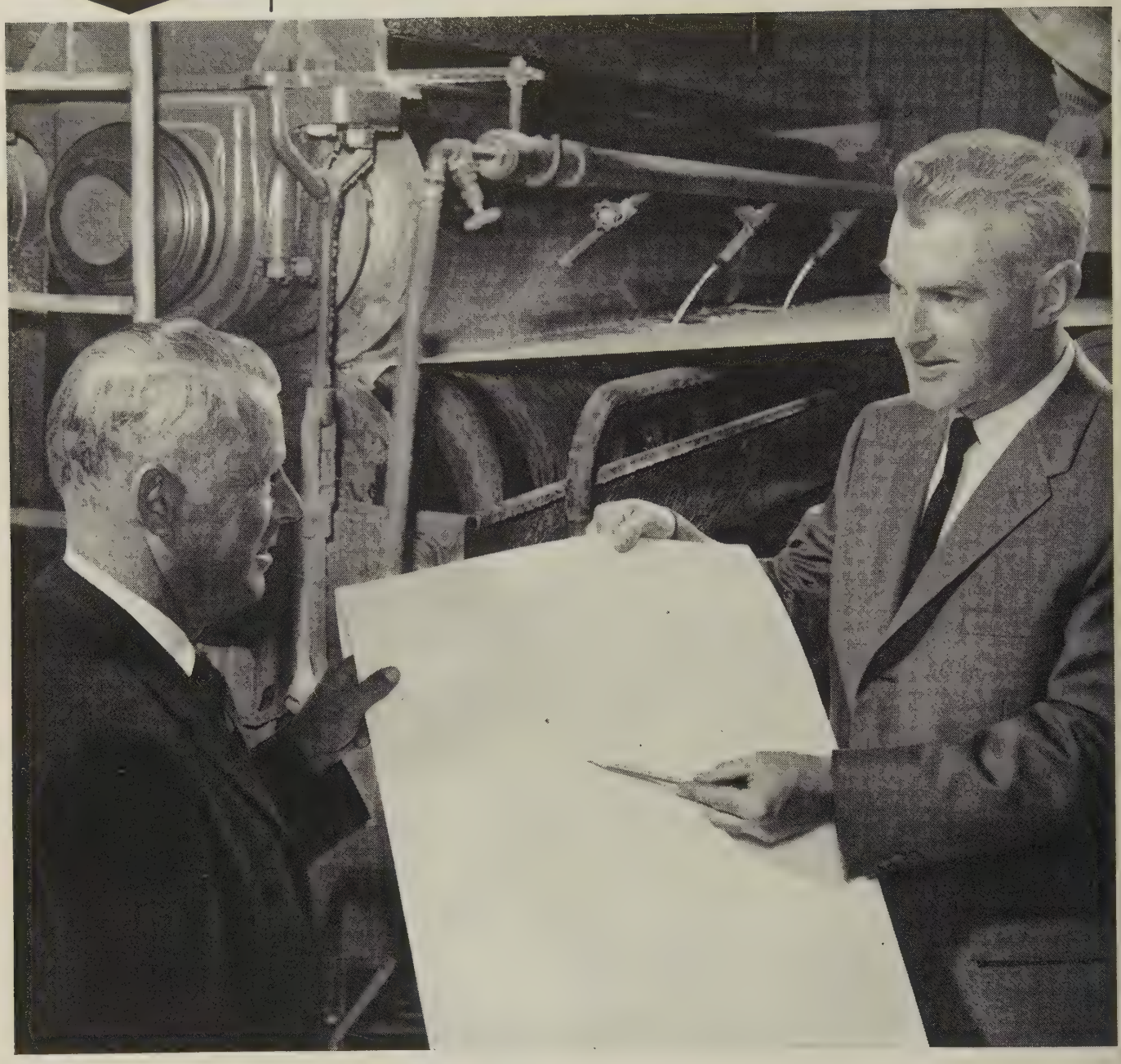
This installation points up the virtue of close cooperation between manufacturer, consulting engineers and instrument supplier. Engineers from Ecusta, Chas. T. Main, Inc. and Taylor Instrument Companies worked as a team to produce a quality control system for No. 10 of which Ecusta is justly proud.

• • •

On your next instrumentation problem, why not sample these established sources of lasting satisfaction—Taylor's broad experience with paper mill problems, our teamwork with your technical forces and your consultants, and the finest in instruments whether pneumatic or electronic. Calling in your Taylor Field Engineer will cost you nothing—it may save you much. If you prefer, write Taylor Instrument Companies, Rochester 1, New York or Toronto, Ontario.

MEAN ACCURACY FIRST

DOW | COATINGS



Paper maker tries Latex for "size"

. . . finds new adhesive permits higher solids content in coatings

In search of a better adhesive for their pigmented size press coatings, a nationally known manufacturer of coated and uncoated paper found the answer in low-cost Dow Latex. Here's their success story!

Modifying their original formula—an all-starch adhesive—with a percentage of Dow Latex, they were able to produce a binder with a lower viscosity. This permitted them to revise their coating formulation to allow for a higher solids content.

The results were gratifying: heavier coatings with better coverage, better printing quality. And the presence of latex in the formula gives the coating unique advantages . . . greater flexibility . . . better water resistance . . . easier calendering . . . higher gloss.

In much the same manner, Dow Latex can give your paper products a quality boost. It's certainly worth investigating . . . contact your nearest Dow Sales Office for more details. Or write THE DOW CHEMICAL COMPANY, Dept. 2322FC9, Midland, Michigan, for complete technical information.

THE DOW CHEMICAL COMPANY • MIDLAND, MICHIGAN

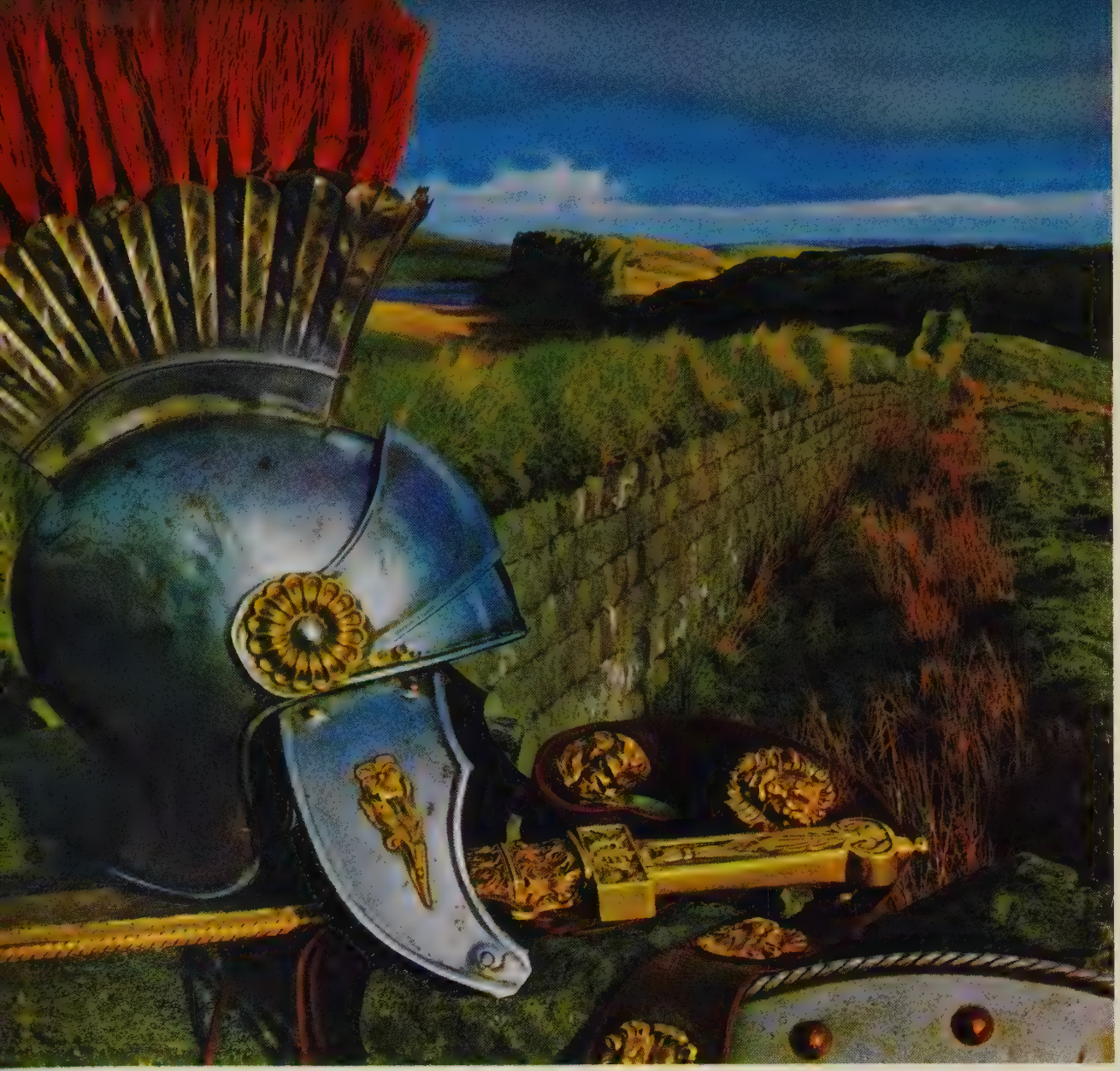


Illustration courtesy of The Curtis Publishing Company

RHOPLEX

Fidelity in Offset... with RHOPLEX

The subtle tones and detail of this illustration, printed by four-color offset lithography, are reproduced in true value because of the paper on which it is printed . . . paper which uses RHOPLEX all-acrylic emulsion as the binder in its coating.

Sharpness of halftone dot definition, freedom from picking, and exceptional varnish holdout are features which appeal to the printer who demands quality of reproduction with high speed presswork.

Papermakers like the ease with which RHOPLEX can be used for on or off-machine coating by trailing blade, air knife or roller coater. They can achieve high coatings weights and still enjoy high speed operation because coatings as high as 65 percent solids still have good handling viscosity when RHOPLEX is the binder.

For full information about RHOPLEX B-15 and RHOPLEX B-60A write for your copy of "RHOPLEX Acrylic Emulsions in Paper Coatings".

RHOPLEX *is a trademark, Reg. U.S. Pat. Off. and in principal foreign countries.*

Chemicals for Industry

ROHM & HAAS COMPANY

THE RESINOUS PRODUCTS DIVISION

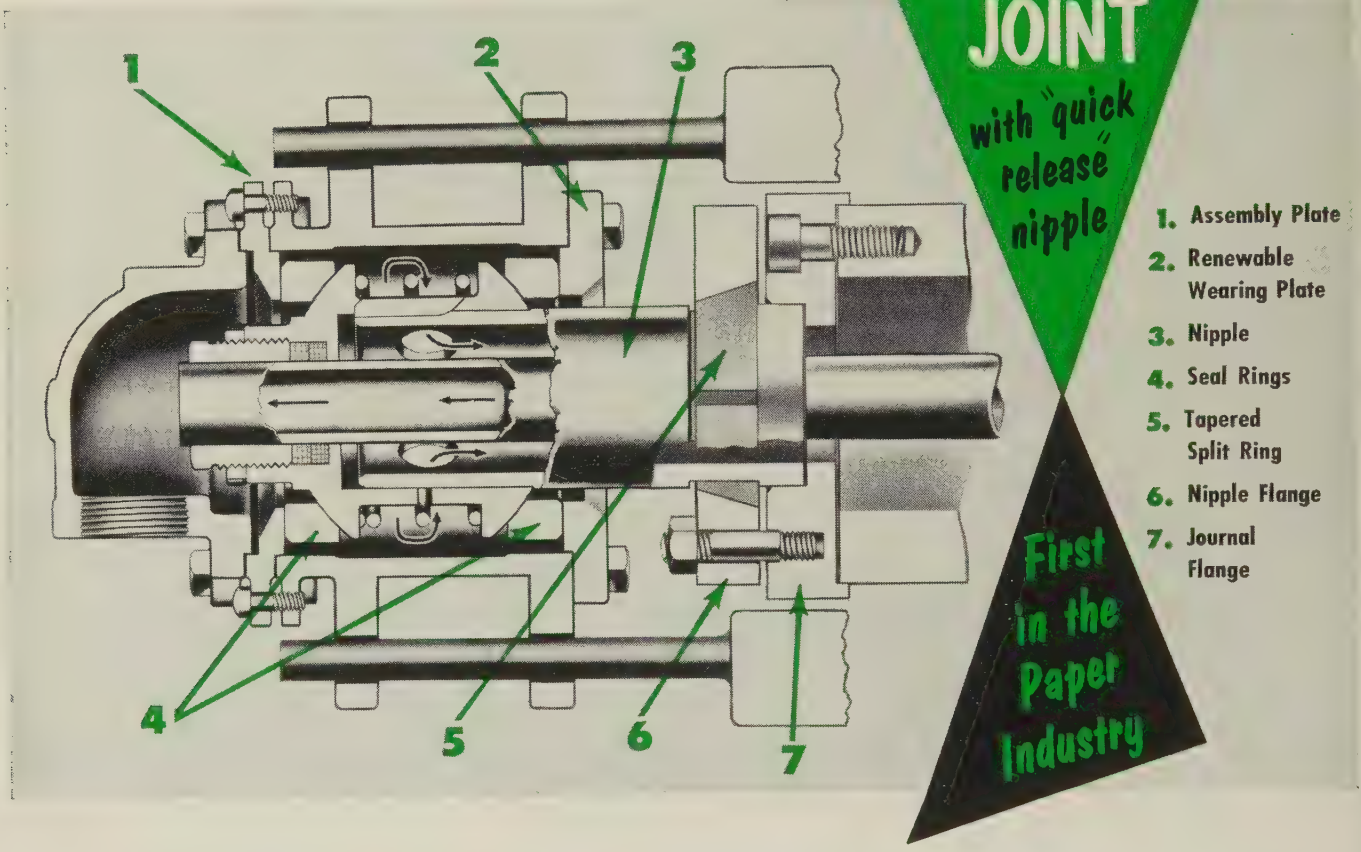
Washington Square, Philadelphia 5, Pa.

This one's got everything

. . . FOR HIGH SPEED PAPER MACHINES WITH ROTATING SYPHON PIPES

SUPER Type L-NARQ JOHNSON JOINT

with "quick release" nipple



1. Assembly Plate
2. Renewable Wearing Plate
3. Nipple
4. Seal Rings
5. Tapered Split Ring
6. Nipple Flange
7. Journal Flange

First in the Paper Industry

Simple, Time-Proved Design—Has the famed packless seal perfected by Johnson, needs no lubrication or adjustment. Internal assembly has just a few simple parts, which means fewer breeding places for trouble, easier field servicing. Assembly plate provides ready access to syphon pipe.

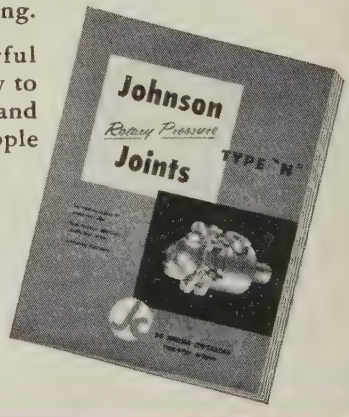
SUPER Construction—Teams "Green Streak" seal rings, of much harder and denser structure, with special Ni-Chrome plating on wearing surfaces. Result: Greatly improved friction characteristics and much longer service life.

Floating Action—Simple rod supports carry all the weight of the Type L body and connections—permit the rotating assembly to "float" freely inside. Can be adapted for machines with open or enclosed gearing.

Quick Release Nipple—Utilizes powerful wedging action to lock nipple securely to journal. Greatly simplifies installation and removal—ends danger of damaging nipple with heavy wrenches.

Johnson Joints come in types and sizes for all needs. Our offer still stands—try a pair in your mill for 90 days without obligation.

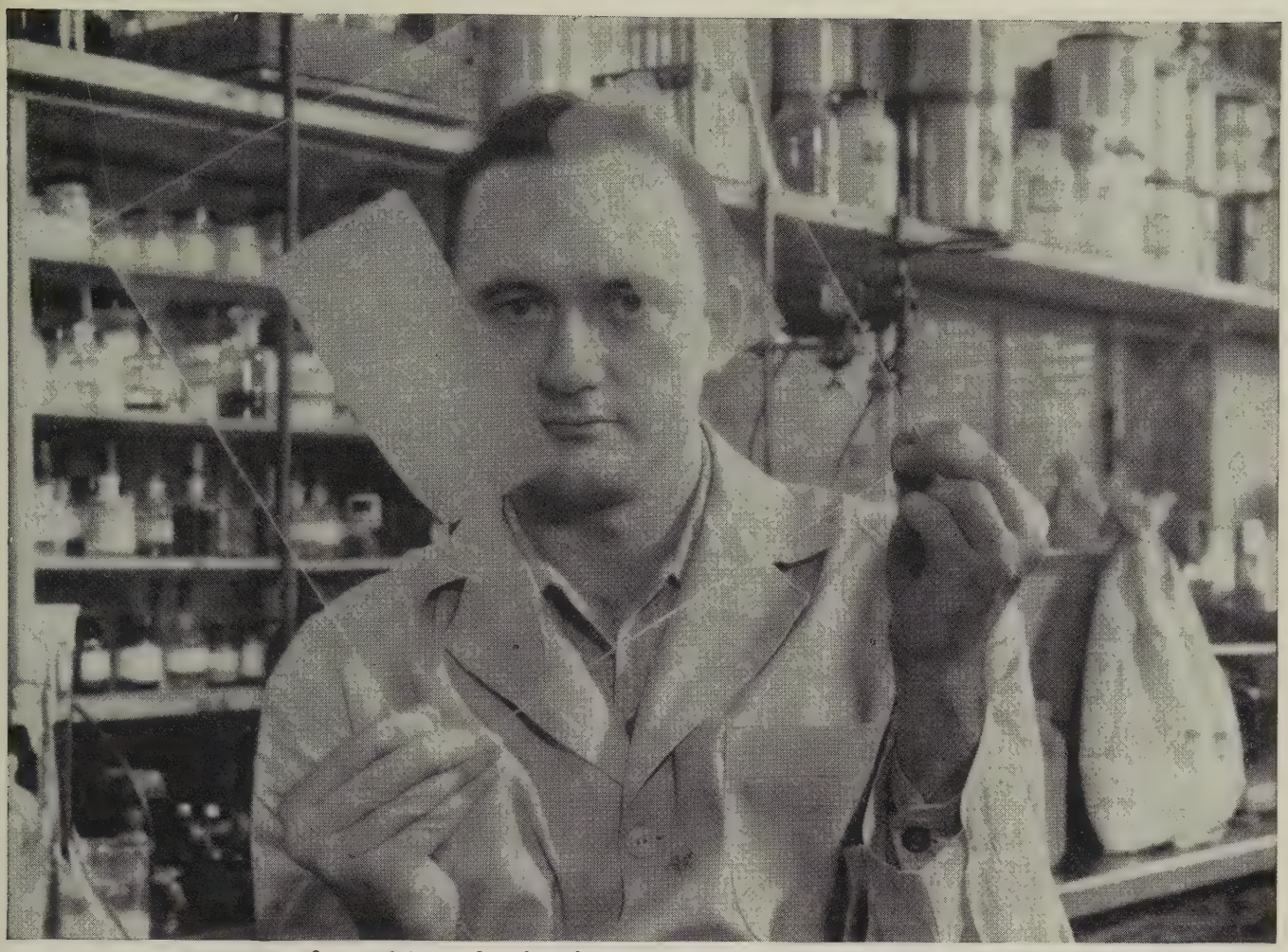
For full information on the Type L-NARQ write for copy of Bulletin N-2002.



THE JOHNSON CORPORATION

843 WOOD STREET • THREE RIVERS, MICHIGAN

Rotary Pressure Joints • Direct Operated Solenoid Valves



Smears of size on glass show clarity of Ten-O-Film, right, almost invisible, compared with conventional starch, left.

Sure way to CLEAR, UNIFORM COATING

The highest degree of coating quality can be achieved with the new, improved Ten-O-Film starches.

These new starches possess unexcelled clarity, uniformity and film-forming properties. They are available in a series of fluidities to meet your exact requirements. Because of their high degree of viscosity stability at high solids, Ten-O-Film starches reduce setback and maintain adhesive strength, assure good performance. At lower concentrations Ten-O-Film starches assure superb structural film continuity and excellent results.

For technical assistance in selecting the TEN-O-FILM starch best suited to your needs contact our nearest sales office or write direct.

TEN-O-FILM® starches

Fine products for the Paper Industry: EAGLE® • FOXHEAD® • GLOBE® • TEN-O-FILM® and CLARO® starches. GLOBE® dextrines and gums.

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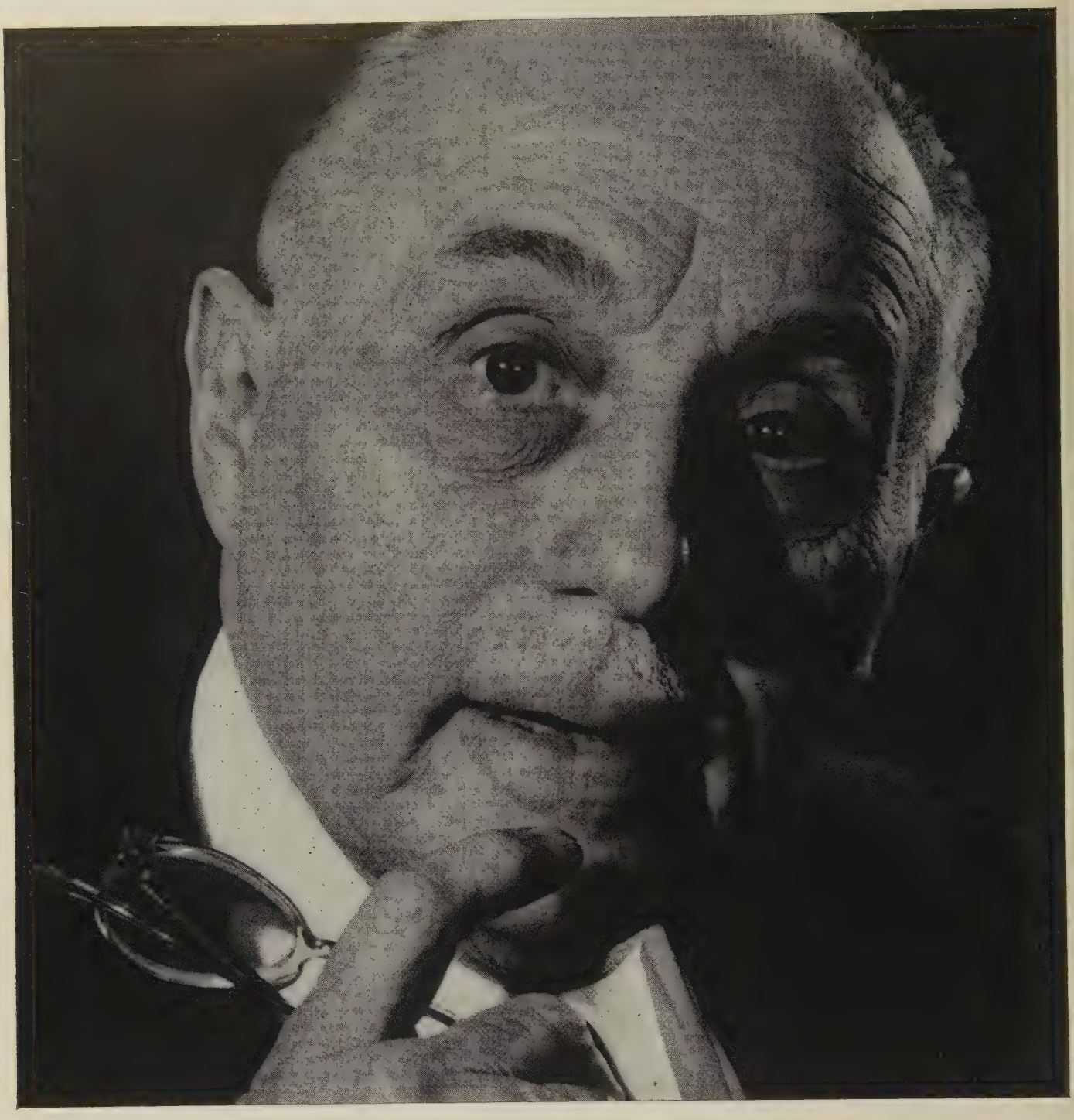

"You can't top TiO_2 as a source of brightness, whiteness and opacity for paper products. We've tried—it can't be done. I don't think you can top Du Pont 'Ti-Pure'* among TiO_2 pigments, either. It's always high in quality, uniform and delivered promptly. Du Pont Technical Service is good, too . . . they had a man working on our beater additions for two days straight once. I understand Du Pont is expanding production facilities for 'Ti-Pure' to take care of future increased demands. Good!"

(*Based on an actual conversation between a Du Pont Representative and a customer.*)

▶ Talked to *your* Du Pont Pigments Representative lately? He'll be glad to point out the "extras" you get with Du Pont "Ti-Pure" titanium dioxide pigments. Call him today! E. I. du Pont de Nemours & Co. (Inc.), Pigments Department, Wilmington 98, Delaware. In Canada: Du Pont of Canada Limited, P.O. Box 660, Montreal, Quebec.

TI-PURE®
Titanium Dioxide Pigments

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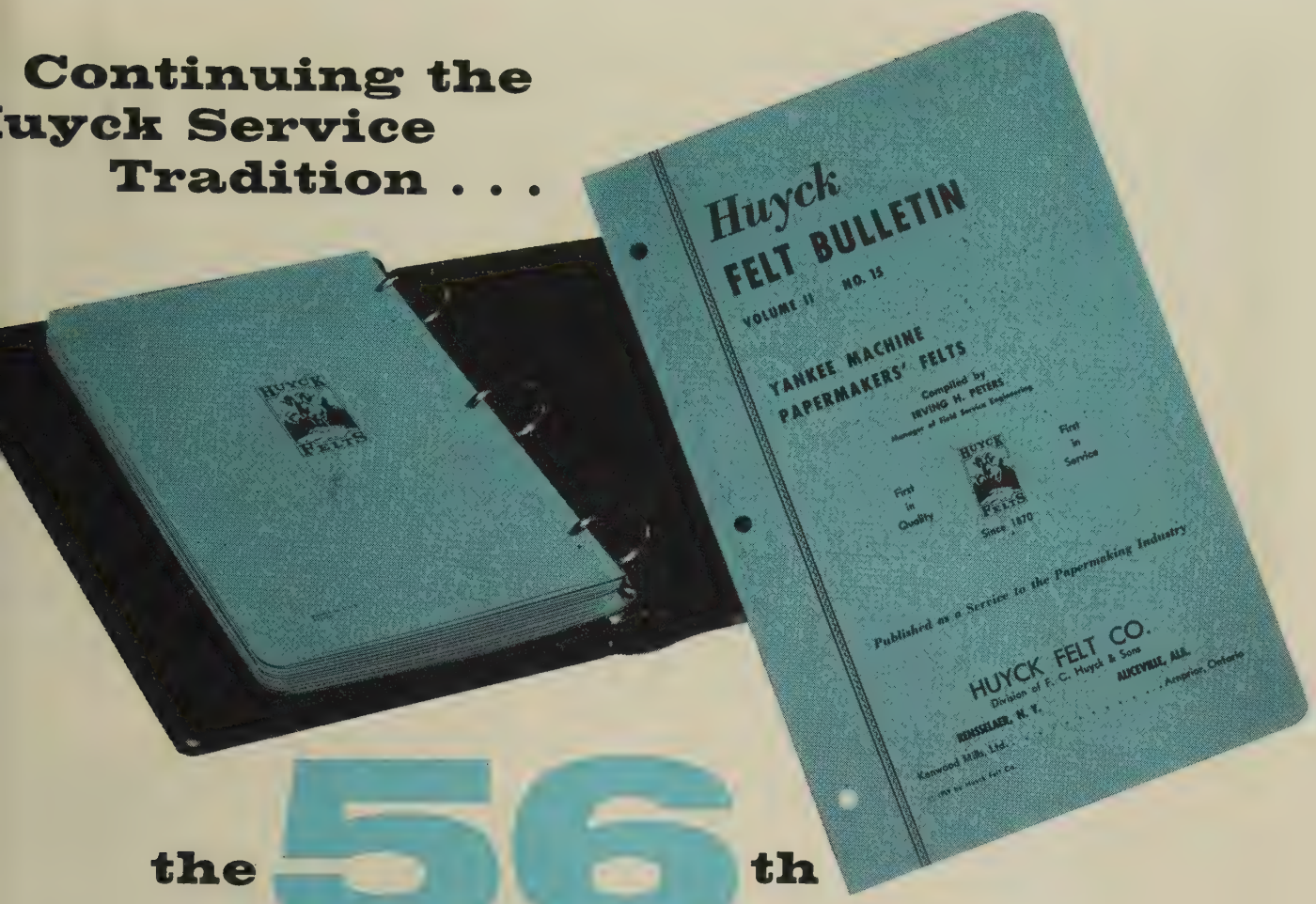
BETTER THINGS FOR BETTER LIVING
. . . *THROUGH CHEMISTRY*

*DU PONT'S TRADEMARK FOR ITS TITANIUM DIOXIDE PIGMENTS

Continuing the Huyck Service Tradition . . .



the 56th . . . in Huyck's series of FELT BULLETINS . . . the most complete, most concise reference library on the application of Papermakers' Felts

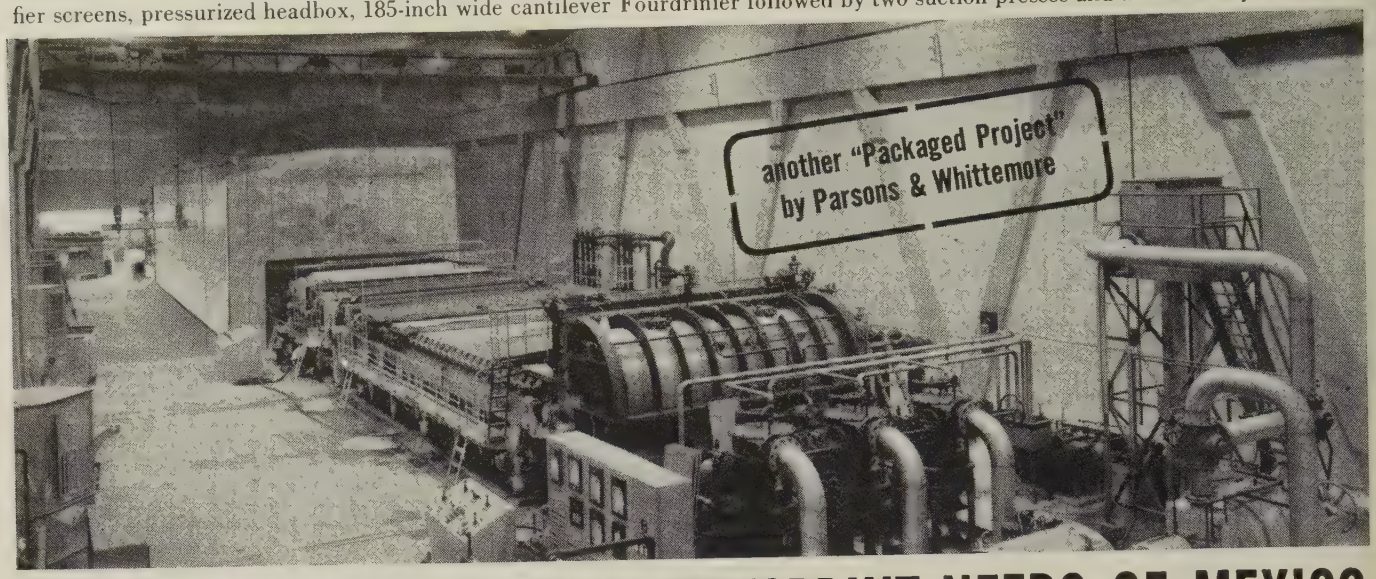
In addition to conducting Felt Workshops and Forums to supply general felt application information, Huyck has published for many years a series of detailed Felt Bulletins that keeps papermakers posted on "what's new" in the use and care of felts. This latest bulletin, the 56th in the series, presents questions and answers concerning practical problems connected with **Yankee Machine Papermakers' Felts** and continues the Huyck Service Tradition of providing operating information of current interest for better felt performance.

Huyck Felt Co.,
Rensselaer, N. Y.;
Aliceville, Ala.;
Division of F. C. Huyck & Sons
In Canada: Kenwood Mills Ltd.,
Arnprior, Ontario.

HUYCK FELTS ★ INDUSTRIAL FABRICS

RST IN QUALITY • FIRST IN SERVICE SINCE 1870

Fabricas de Papel Tuxtepec's modern air-conditioned pulp and paper mill is laid out for straight line flow of materials from the woodyard to the finished newsprint shipping dock. Shown here is the Black-Clawson newsprint paper machine with its three Selectifier screens, pressurized headbox, 185-inch wide cantilever Fourdrinier followed by two suction presses and a 39-unit dryer section.



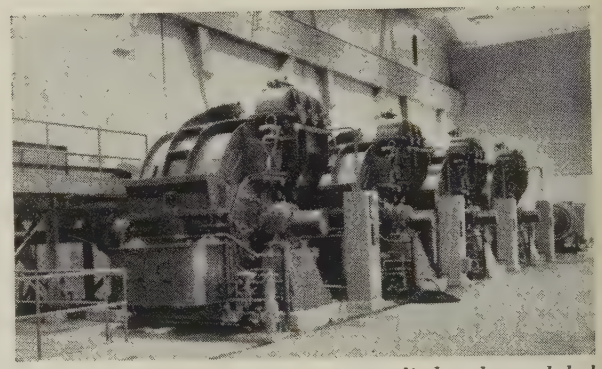
NOW PRODUCING HALF THE NEWSPRINT NEEDS OF MEXICO

Mexico's first newsprint mill, which started operation at the end of 1958, is now producing 100 tons of newsprint daily from local pine. The entire $15 million project—planning, financing, plant equipment and initial operation of the mill—was handled by the Parsons & Whittemore-Lyddon Organization. The mill was designed by Sandwell & Co., Ltd. on behalf of Fabricas de Papel Tuxtepec S.A.

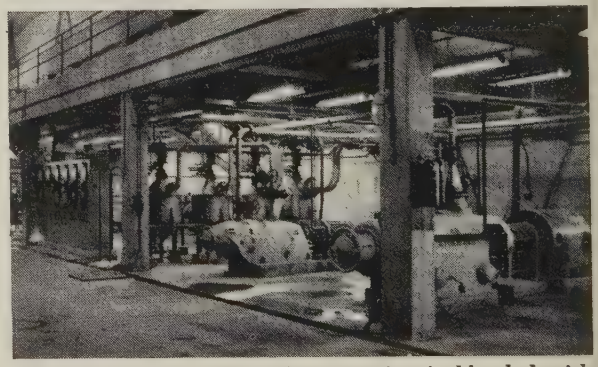
With more than 100 years' experience in pulp, paper and machinery...with manufacturing facilities on three continents...and with engineering and financial associates throughout the world, we can render *any* or *all* of the following services:

1. ***Conduct*** a thorough technical and economic field survey and prepare a complete appraisal report.
2. ***Assist*** the local group in financing the project.
3. ***Design*** the complete project in cooperation with the world's leading consulting engineers.
4. ***Produce*** and deliver all pulp and paper making machinery and other equipment which is not locally available.
5. ***Supply*** skilled personnel to ensure efficient operation until the local staff has been trained.
6. ***Cooperate*** in procurement of foreign raw materials and sale of the mill's output on world markets through our own branches.

For full information on our services, write our nearest office.



All equipment for Tuxtepec was supplied under a global contract by the Parsons & Whittemore Organization. Four Roberts Grinders, each with a 3,000 horsepower, 257 rpm motor, were manufactured by The Black-Clawson Company, Pandia Division, Hamilton, Ohio.



Groundwood pulp from Mexican pine is blended with chemical pulp. Black-Clawson Hydrafiners and a No. 6 jordan prepare the stock for the paper machine.

THE PARSONS & WHITTEMORE/LYDDON ORGANIZATION

World leaders in the development of pulp and paper mills for the use of local fibers

18-19 Savile Row, London W. 1, England

5 Rue Jean Mermoz, Paris 8°, France

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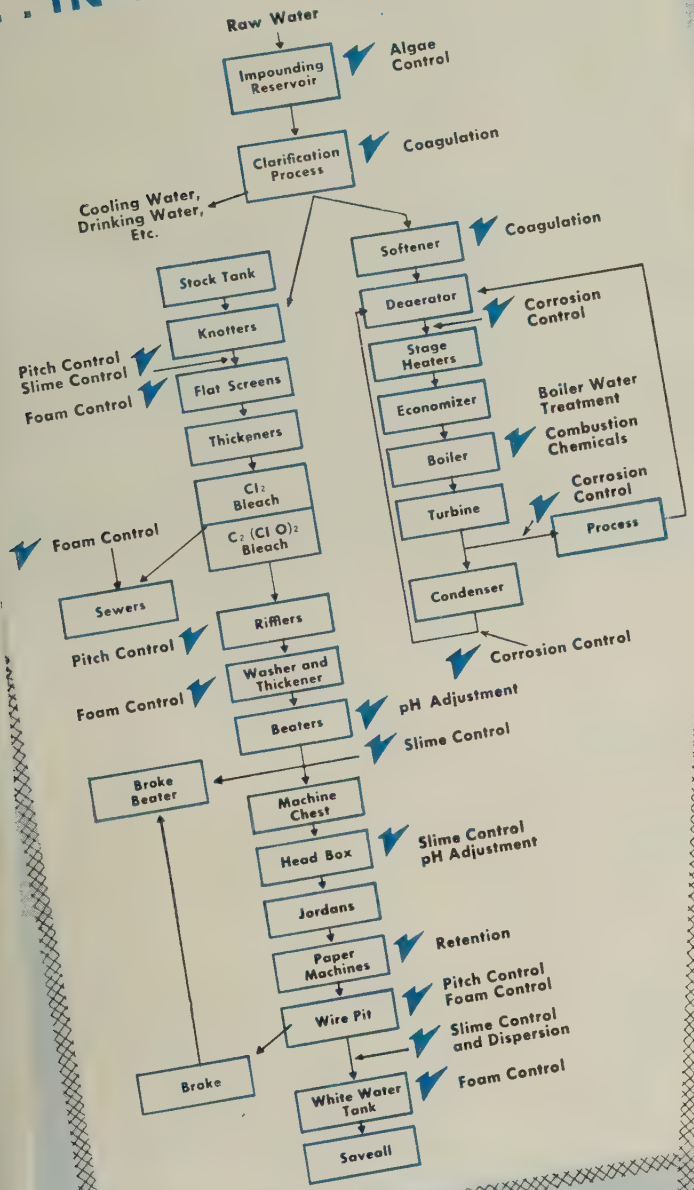
HERE IS THE

COMPLETE *Nalco* PROGRAM

for Better Products at Lower Cost

. . . IN A TYPICAL MILL



NALCO has the *widest variety* of formulas to assure results in your mill, too. Here are some examples . . . representative Nalco products listed under headings which indicate the job they do. And the chart at left tells you where they are used. Your Nalco Representative will tell you more about them—or write direct to Nalco.

ALGAE CONTROL
Nalco Cuprose, Nalco 21-S, Nalco 201, Nalco 234, Nalco 236, Nalco 237

COAGULATION
Nalco 600, Nalco 650, Nalco 680

COMBUSTION CHEMICALS
Nalco 155, Nalco 156, Nalco 156-C

FOAM CONTROL
Nalco 71, Nalco 71-B, Nalco 71-C, Nalco 71-D5, Nalco 71-K, Nalco 72

CORROSION CONTROL
Nalco 19, Nalco 35, Nalco 353, Nalco 918

pH ADJUSTMENT
Nalco 680

PITCH CONTROL
Nalco 818

RETENTION AND CLARIFICATION
Nalco 600, Nalco 650, Nalco 680

SLIME CONTROL AND DISPERSION
Nalco 21-S, Nalco 21-M, Nalco 201, Nalco 234, Nalco 236, Nalco 237

BOILER WATER TREATMENT
Nalco 75, Nalco 718, Nalco 728, Nalco 752

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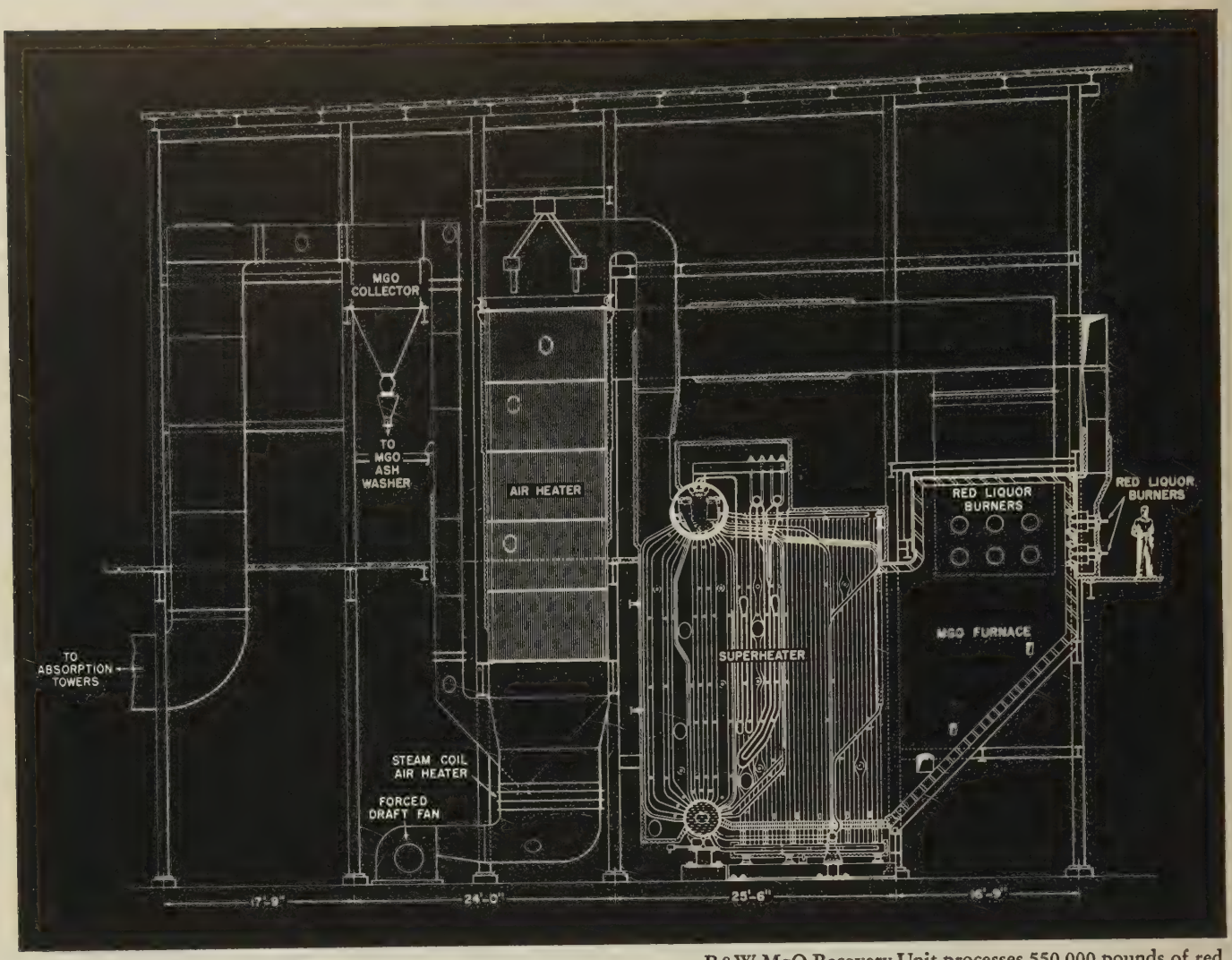
THE *Nalco*® SYSTEM . . . Serving the Paper Industry through Practical Applied Science

Nalco CHEMICALS

61 A



B&W MgO Recovery Unit processes 550,000 pounds of red liquor solids per day, generates 115,000 pounds of steam per hour at 850 psi and 825 F at the superheater outlet.

MgO CUTS CHEMICAL COSTS

B&W MgO Pulping and Recovery Process Sets New Standards for Chemical Recovery at Weyerhaeuser Sulfite Pulp Mill

At Weyerhaeuser Timber Company's new sulfite Pulp Mill, Cosmopolis, Washington, the B&W MgO Pulping and Recovery Process has established an impressive record of efficient and economical chemical recovery. *Chemical make-up for the complete process is only 20 pounds of MgO and 50 pounds of sulfur per ton of unbleached pulp produced.*

Low chemical make-up and high steam generation reduce pulping costs. For this reason, the B&W MgO Recovery Process has rightfully earned the acceptance of the Pulp Industry. Start-up of the fifth MgO pulping and recovery installation during 1959 will increase pulp production by this process to 650,000 tons per year.

Talking over your chemical and heat recovery problems with B&W can be your first step toward reducing pulping costs and increasing profits. Why not write today for complete information? The Babcock & Wilcox Company, Boiler Division, Barberton, Ohio.

P-821

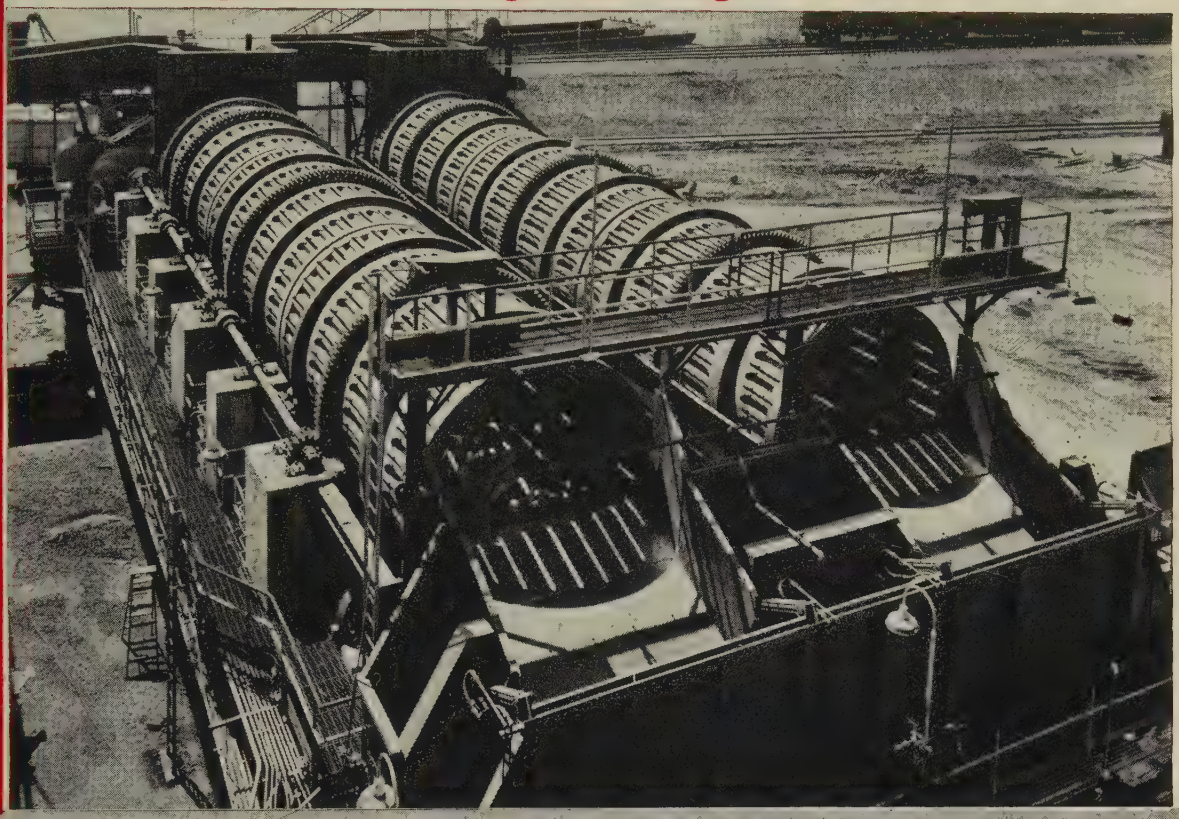
B&W

THE BABCOCK & WILCOX COMPANY

BOILER DIVISION

The "Weldrum" Barker...

Progressive Engineering in action



These 67½ feet three-section "Weldrum" Barkers are installed at Bowaters Southern Paper Corporation, Calhoun, Tennessee.

Reducing the customer's cost of drum barking wood . . .

. . . is the primary purpose behind the design of every "Weldrum" Barker.

This continuing research may be called progressive engineering and it is done in full co-operation with the engineering staffs of our major pulp and paper producers.

Actually, our program of modifying and improving the "Weldrum" Barker has produced unparalleled savings in drum barking costs.

Four basic models cover practically all drum barking operations:

- *12′ by 45′ two-section drum for most soft woods*
- *12′ by 45′ two-section drum with solid plate inlet section for extra washing or frozen wood*
- *12′ by 67½′ three-section drum for highly resinous wood*
- *12′ by 67½′ three-section drum for hard woods*

For specific information and detailed estimates, contact your nearest Ingersoll-Rand office or write direct to Canadian Ingersoll-Rand Co. Limited, 620 Cathcart Street, Montreal, Canada.

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Staley's BETTER PRODUCTS FOR THE PAPER INDUSTRY

WAX-PICK TESTS PROVE...

Staley's new Stadex® Dextrin 140 gives greater binding strength in higher-solids coating colors

Higher adhesive quality! Now you can use high-solids coatings of low viscosity *without sacrificing surface strength.*

Staley's new improved Stadex Dextrin 140 solves the problem of poor binding strength in high-solids coatings once and for all. Stadex Dextrin 140 is a high-solids clay coating adhesive with excellent stability, viscosity and binding strength characteristics. Used in combination with Stayco® (oxidized) starches, Stadex Dextrin 140 produces a coating of high adhesive quality.

Long a standard in the paper industry, Stadex 140, like all Staley dextrins, is now made by a brand new dextrinization technique that guarantees you even better adhesives of top quality, minimum color and unparalleled uniformity.

Why not get the full story on Stadex 140 for yourself now?

See your Staley representative or write us for details today.

Staley's®

A. E. Staley Mfg. Co., Decatur, Ill.

Branch Offices: Atlanta · Boston · Chicago · Cleveland · Kansas City
New York · Philadelphia · San Francisco · St. Louis

LET HART
BRING YOU THE SOLUTION TO YOUR MILL PROBLEMS

With chemicals for every phase of paper mill operation. Uniform products designed for mill use—technical assistance in application.

Together, they can provide you with a dependable source for the chemicals and service you need in processing your pulp and paper.

We suggest that you get in touch with a Hart Products representative to discuss the matter.

HART PRODUCTS BASED ON RESEARCH

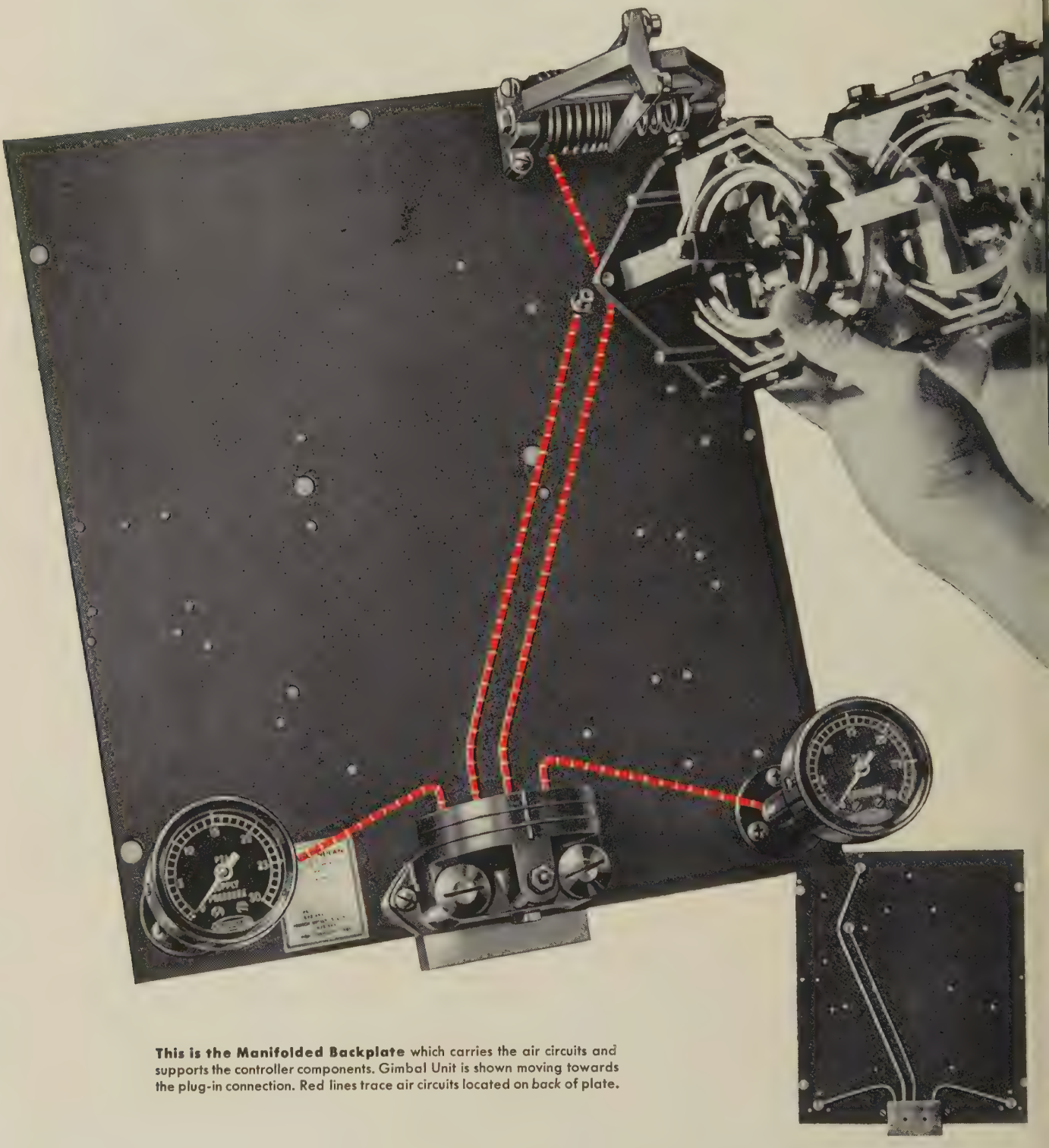
the Hart Products Corporation

1440 BROADWAY, NEW YORK 18, N. Y.

Works and Laboratories, Jersey City, N. J.

Masoneilan 60000 Series

Controllers Feature Plug-in Connections, Manifolded Air Circuits



This is the Manifolded Backplate which carries the air circuits and supports the controller components. Gimbal Unit is shown moving towards the plug-in connection. Red lines trace air circuits located on back of plate.

The manifolded backplate . . .

- **Eliminates exposed tubing and pressure fittings**
- **Protects air circuits**
- **Provides base for mounting plug-in subassemblies**
- **Permits easy access to operational units for interchange and servicing**

Masoneilan 60000 Series Pneumatic Controllers are designed for utmost simplicity and ease of servicing, with a big plus in ready interchangeability of operational units — gimbal unit, nozzle, proportional unit, reset unit and gauges — by means of plug-in O-ring connections. The manifolded backplate provides a rigid base for components; protects air circuits; eliminates exposed tubing and pressure fittings; avoids leakage. Pilot manifold block allows quick, easy connection of manual control unit.

Result: An instrument offering maximum simplicity and accessibility.

This is only one of the design features which have given the 60000 Series such a fine reputation for dependable, accurate performance; versatility in application; and minimum attention. Complete details available from the nearest Mason-Neilan representative or write

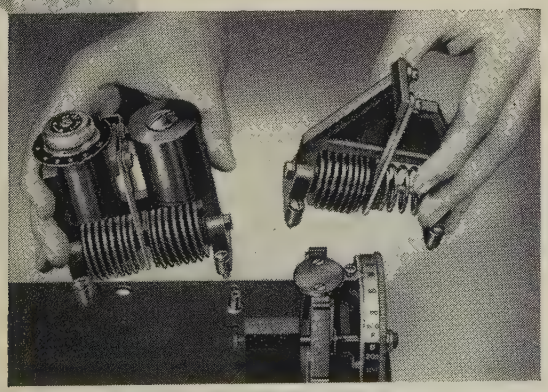
MASON-NEILAN

Division of Worthington Corporation

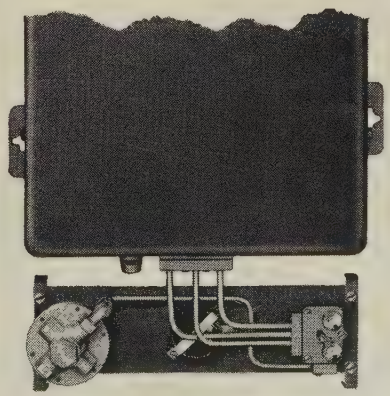
47 NAHATAN STREET, NORWOOD, MASSACHUSETTS

District offices or Distributors in principal cities in U. S.

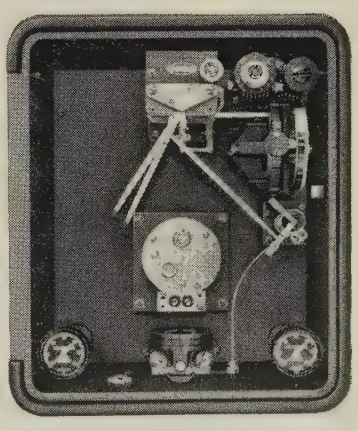
In Canada: Division of Worthington (Canada), Ltd., Mason-Neilan



Proportional and Proportional-Reset Units are easily and quickly interchangeable.



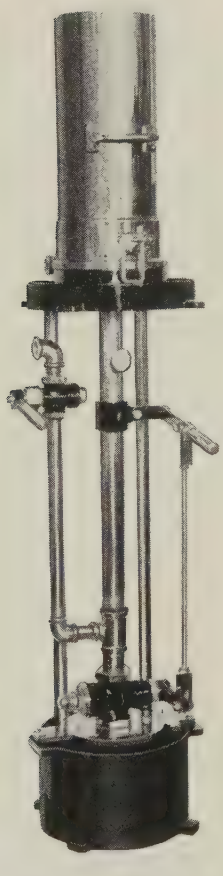
Manual Control Subpanel. Four position transfer switch connects to pilot block by tubing manifold. Operator can select manual, start-up, test, service, and automatic control.



Interior View of Controller shows the accessible, easy-to-service design. There is plenty of room to work; interchangeable components provide trouble-free operation.

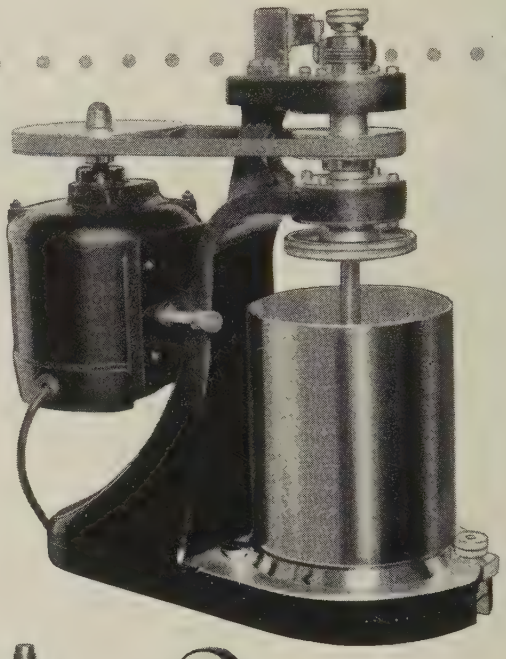
TAPPI STANDARD

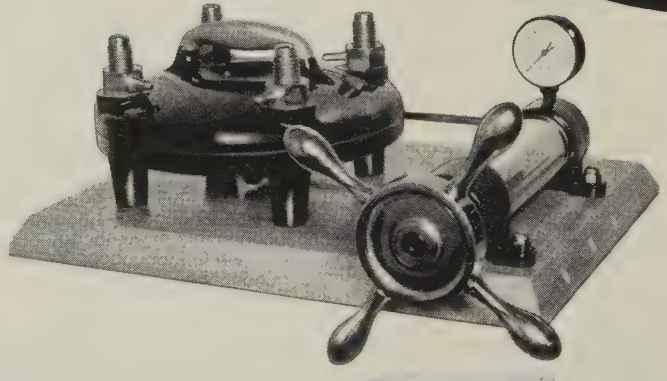
Pulp Testing and Sheet Making Apparatus



TAPPI
Sheet Making
Machine

TAPPI
Disintegrator





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AUTOMATIC COUCHING AND PRESSING EQUIPMENT
as developed by Hermann Mfg. Co.

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THE HERMANN MANUFACTURING CO.
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Let your containers get and hold more business

by using the RIGHT PACKAGING ADHESIVE

from Borden

Containers that win and hold more business must be right—down to their seams. That means using the right Borden adhesive for each bonding requirement—bags, cases, cartons, cups, films, foils, labeling, laminates.

Borden know-how is available to you through its technical service staff and laboratories to help you select the best adhesives for your application.

Get your copy of Borden's new Adhesive Index. It's packed with helpful hints. Mail the coupon to: The Borden Chemical Company, Dept. PF-69, 350 Madison Avenue, New York 17, N. Y.

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Borden **Chemical**

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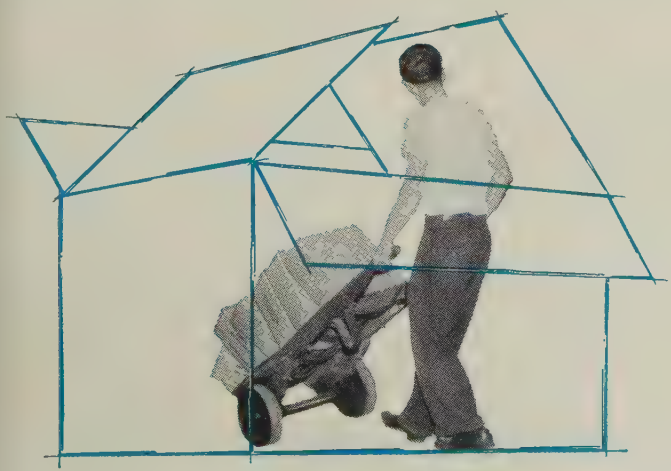

The Borden Chemical Company
350 Madison Avenue
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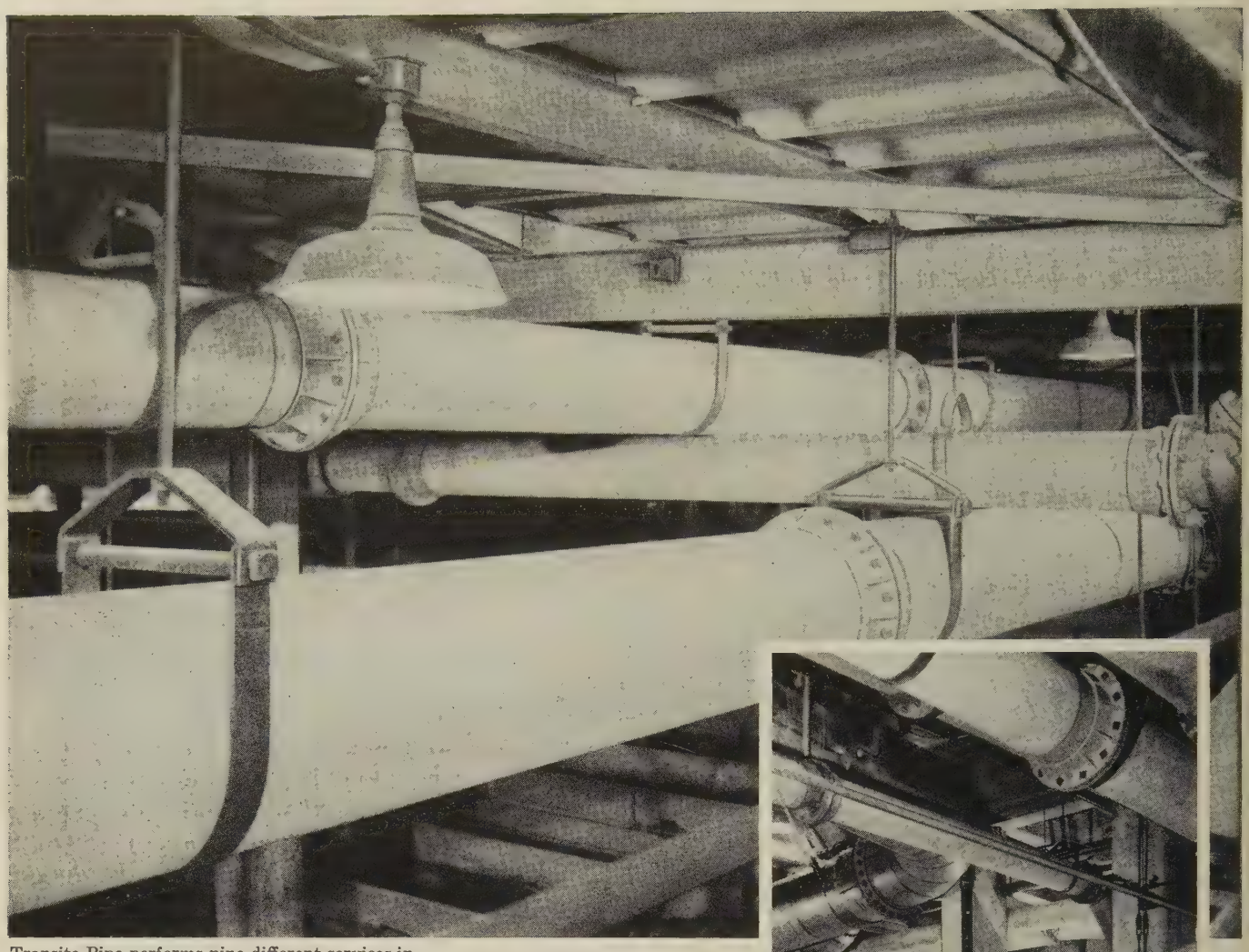
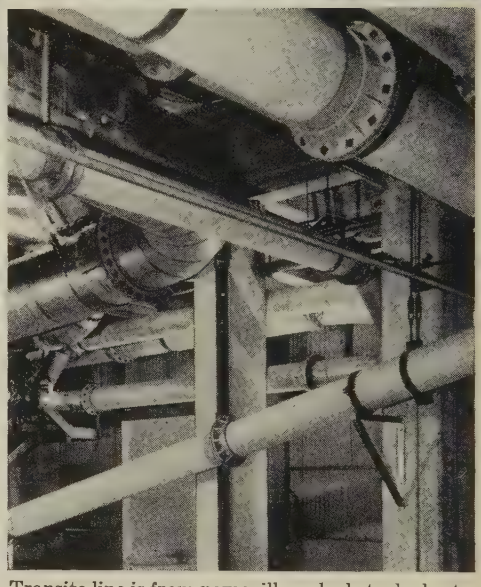


Transite Pipe performs nine different services in the St. Regis Company's Jacksonville Plant.



Transite line is from new mill washed stock chest to old mill washed stock chest.

Transite Pipe helps St. Regis work giant new "Seminole Chief" at full capacity!

Chief Engineer, L. C. Crowder, tells of experience with Transite . . . how its flow capacity and "cleanliness" determined its selection

Almost by the day, we see new reports on the added economy and performance that Transite® Pipe brings to water and stock lines. One such report comes to us from the St. Regis Company's Jacksonville Plant:

"When we recently installed the Seminole Chief, one of the largest capacity machines in the industry," says Mr. Crowder, Chief Engineer, Pulp & Paper Division, "production jumped from 300 to 1300 tons per day. And water capacity and stock cleanliness became more important than ever.

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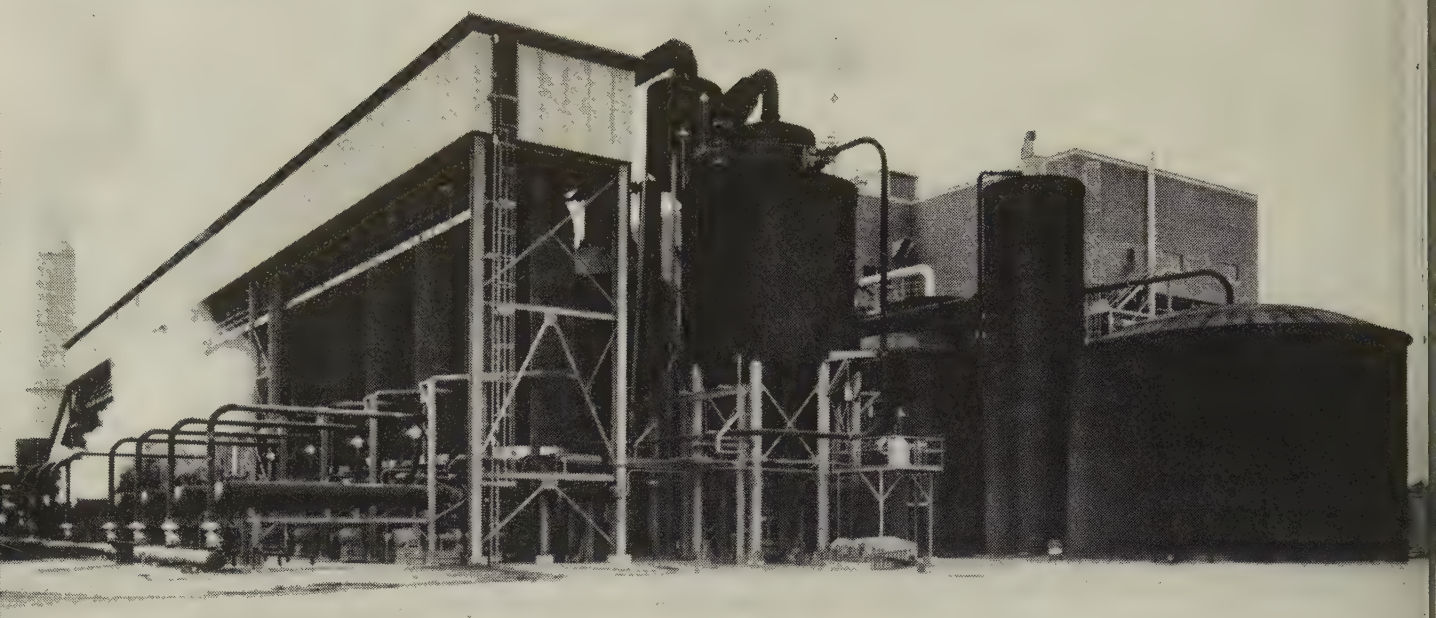

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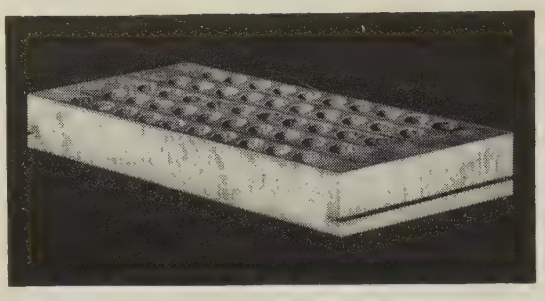

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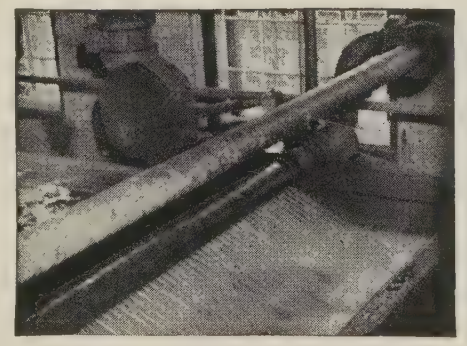

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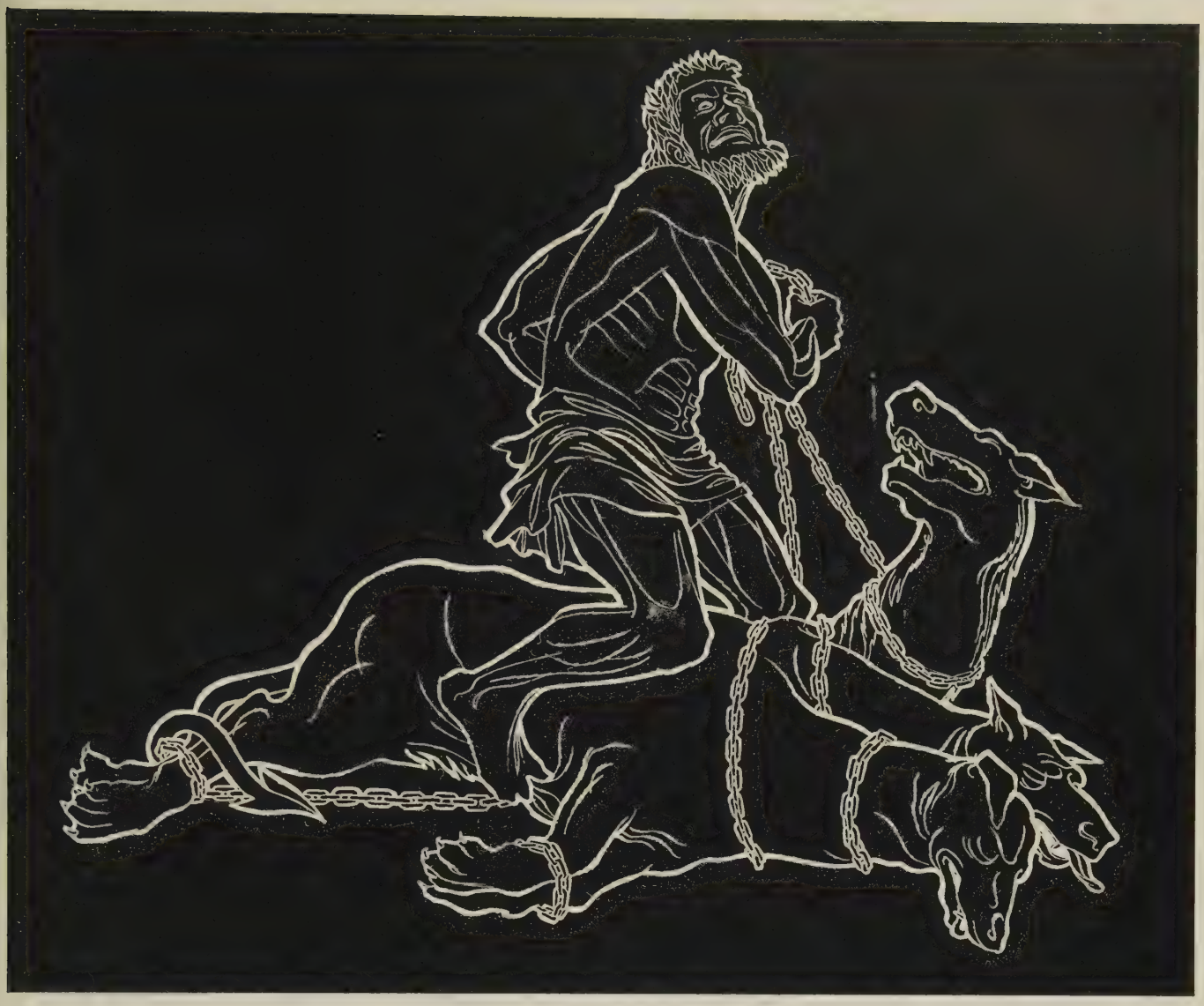

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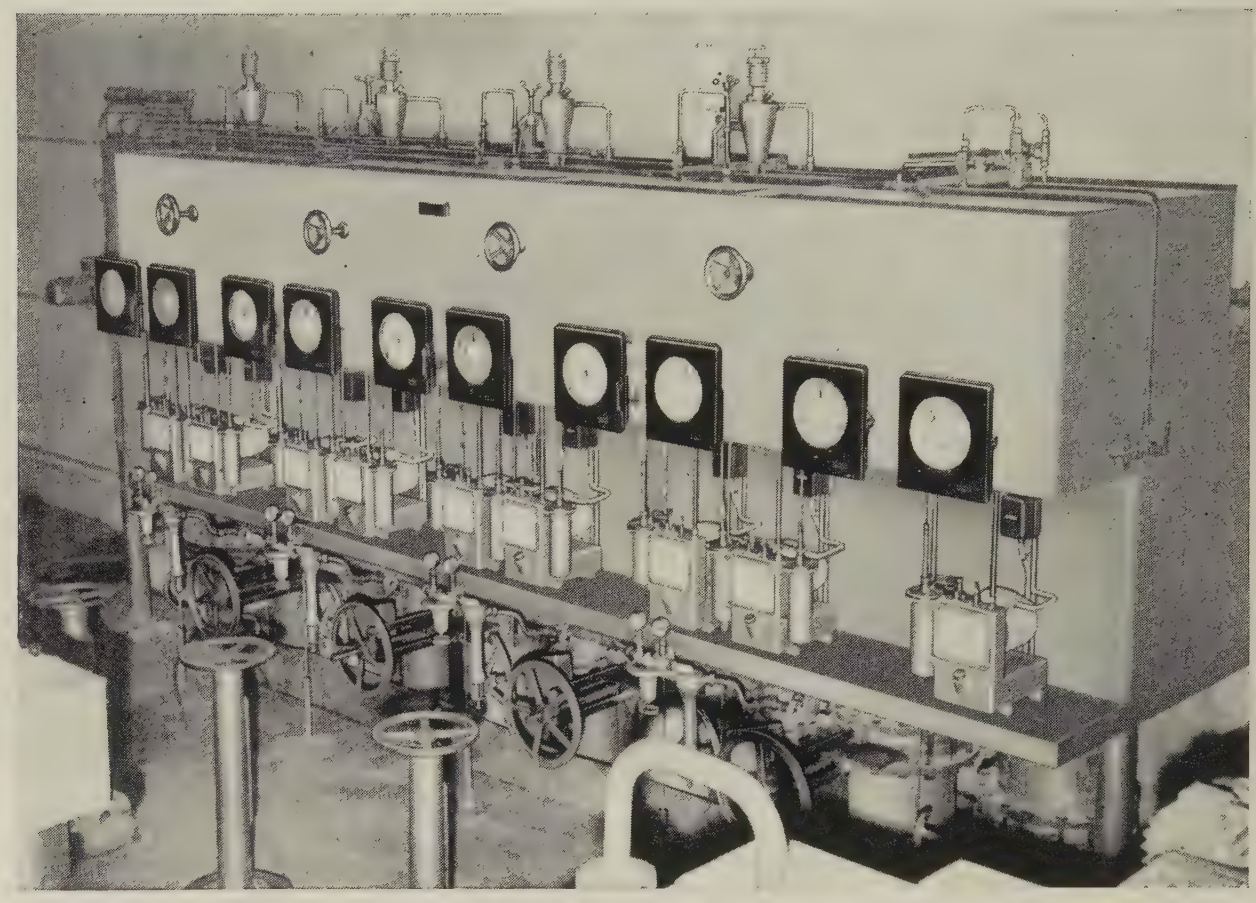

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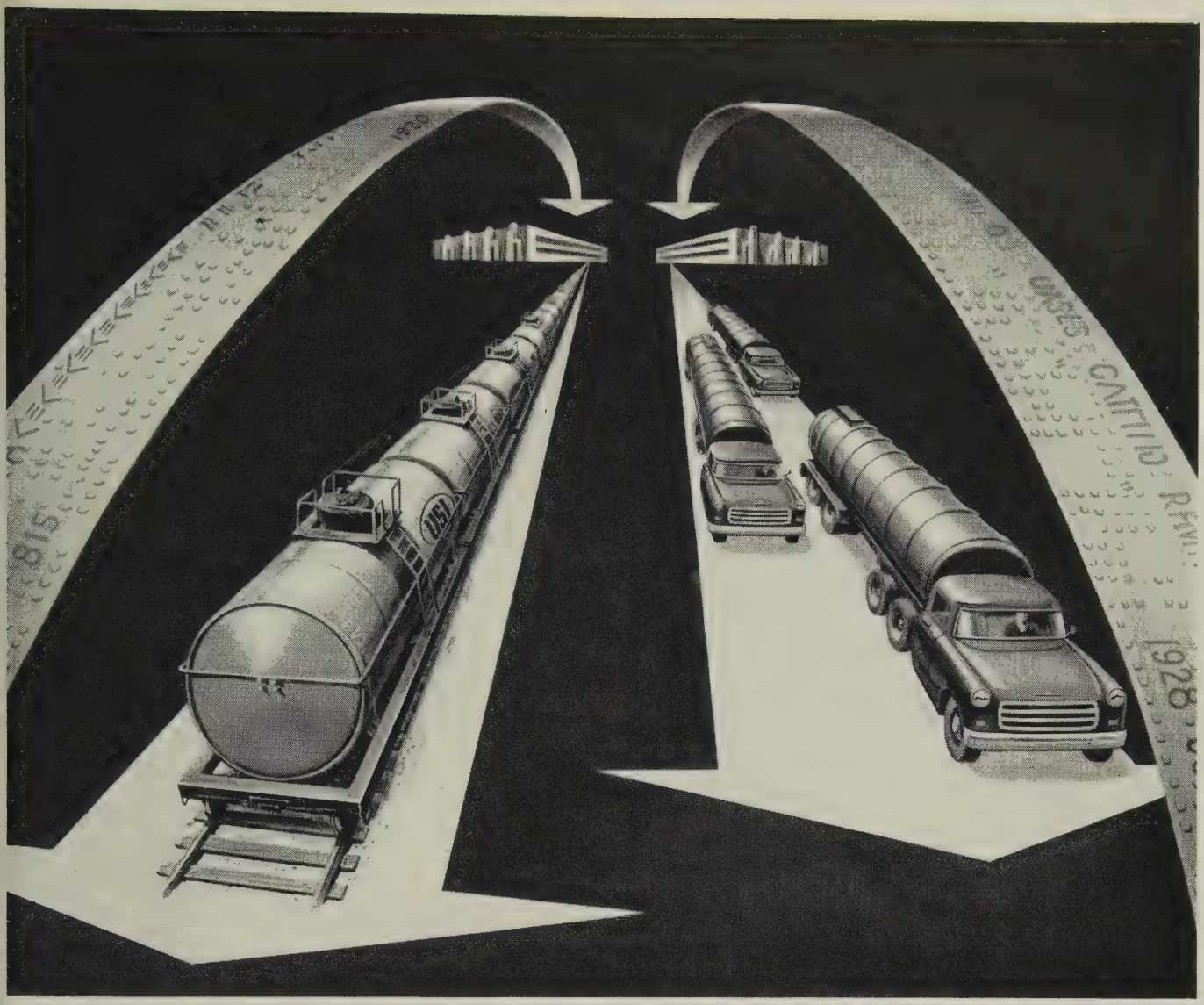

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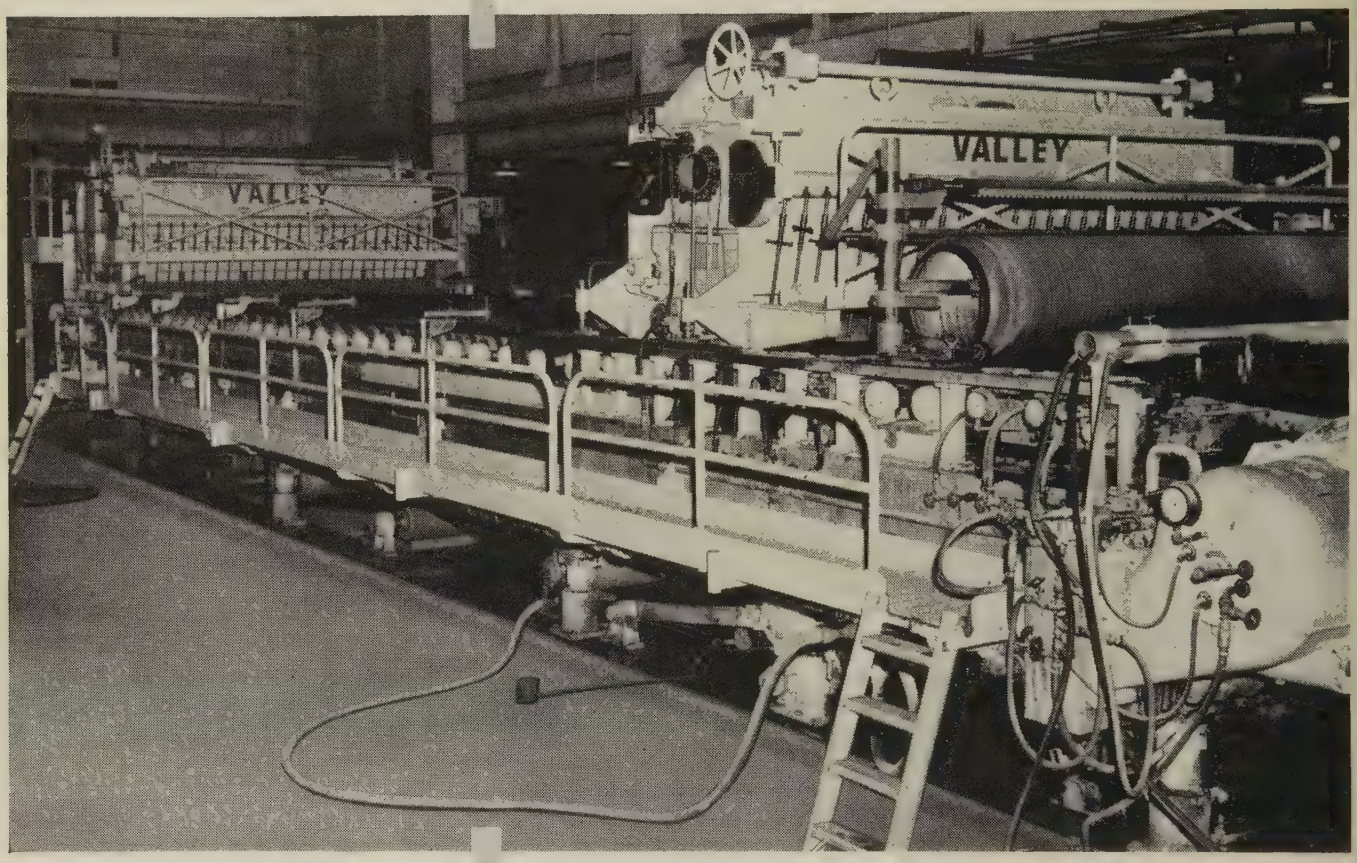

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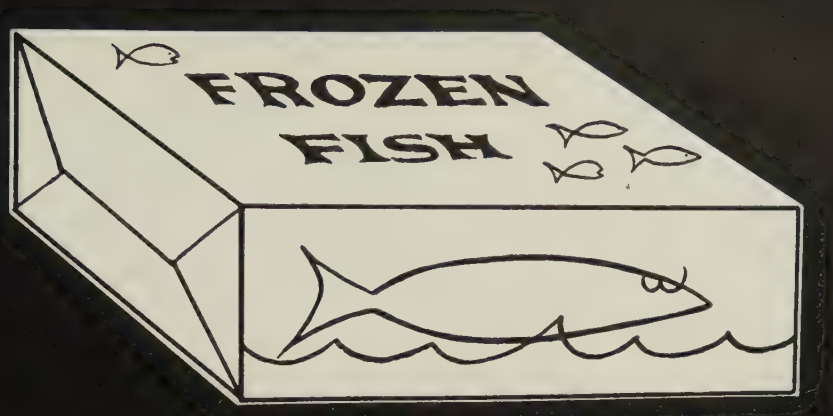

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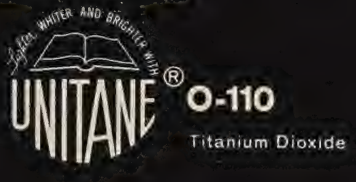

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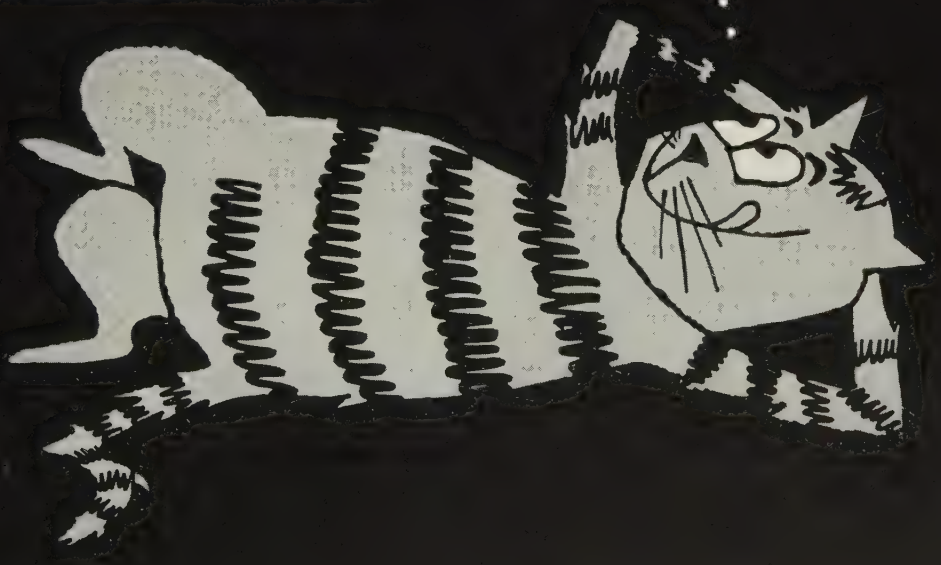

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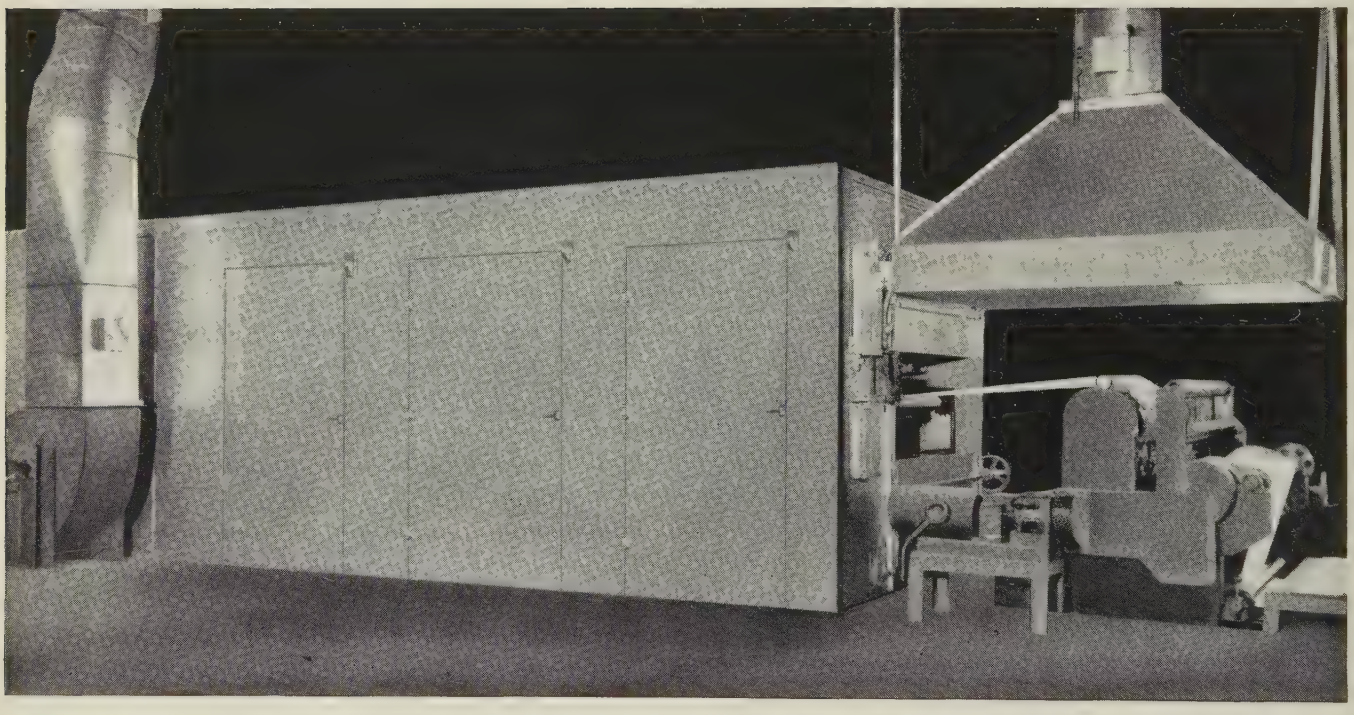

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The Flow Properties of Paper Pulp Stock

IV. The Effect of Fittings on Piping Systems Carrying Pulp Slurries

RICHARD E. DURST

The pulp flow system at the University of Maine was rebuilt during the past year to include 45° taper expanders and reducers, 90° expanders and reducers, 90° long and short radius elbows, and tees on run and side discharge. The fittings were separated by fixed lengths of straight pipe. Pressure taps were installed so pressure drops could be measured over each fitting and each segment of straight pipe. Data were taken for unbleached northern kraft pulp slurries of 2.0, 3.0, and 3.8% consistency at velocities from 1 to 10 ft. per sec. The head loss across the fittings is expressed as equivalent lengths of straight pipe. The principal observations are that the equivalent length of the fittings decreases with increasing consistency of the pulp stock, over the range of consistencies studied, and that the straight sections following fittings show decreasing head loss values with increasing velocity.

THE literature dealing with pulp stock flow reports the results of many investigations (*1–15*) studying the relation of piping materials, pulp types, and pulp consistencies to the frictional head loss suffered as the pulp slurries are pumped through the test sections. The test sections, where they have been defined, have been isolated from situations that would disturb the steady-state flow pattern. The length of calming sections isolating test sections has not been definitely established, and it is probably a function of a number of variables.

The disagreement among the various sets of data reported for pipe friction results from the number of variables that enter into the determination of the head loss. Variation in pulp properties from which the slurries are prepared is probably the major cause of deviation. The progress in establishing a correlation between pipe friction data and properties of pulp slurries resulting from the research sponsored by the Hydraulics Committee of TAPPI* at the University of Maine and the able work done by Victor Head of Fischer and Porter (*12–16*) has removed much of the deviation caused by pulp slurry properties.

The effect of physical arrangement of the equipment, as calming lengths, pipe fittings, length of the sections, static pressure on the stock, and other physical effects as stock temperature and dissolved or entrained gases in the stock have not been defined as fully as have the pulp properties.

The purpose of the work being reported here was to determine the effect of fittings in piping systems carrying pulp stocks. The only previous extensive work reported in the literature was done at McGill University (*17*) investigating the effect of certain smooth bend elbows and welded elbows of two and three segment design for stocks of 2.0 and 2.55% consistency. Unpublished studies of pulp stock piping systems in mills have shown that the average run of straight pipe between major fittings, i.e., elbows, tees, valves, reducers, and enlargers is in the range of 12 to 18 ft. If it is anticipated that fittings disturb the flow pattern for at least 10 and probably as much as 20 pipe diameters beyond the fitting there is very little steady state flow in the average pulp stock flow system. It is therefore apparent that data on the frictional losses in fittings and short straight sections following fittings should contribute to the more accurate design of flow systems and the power requirements for pumping stock through the systems.

Engineering texts and handbooks contain tables and charts for estimating the frictional losses in various fittings for water and other fluids. The values are expressed as equivalent lengths of straight pipe or as coefficients that are used in equations of the type

$$h = k \frac{V^2}{2g} \qquad (1)$$

These sources vary widely in the values given for the fittings reported. The straight pipe sections following fittings are in general considered to have friction characteristics equal to the straight sections in which steady-state flow exists. This assumption would not cause much error where fittings were widely spaced, but could be a factor of considerable importance in systems with numerous fittings and short pipe sections.

EQUIPMENT MODIFICATION

The pulp flow system at the University of Maine (Fig. 1) was modified to provide test sections including some of the more common fittings found in pulp flow systems. Specifically, tees with side discharge and on run, and segmental welded elbows with R/D ratios of 1.4 and 2.0, referred to as short radius and long radius elbows were fitted with pressure taps at the ends of the fittings. The distance between taps on the tees was 15 in. on the center line of the pipe. The distance on the short radius elbow was 13 in. and for the long radius elbow 18 in. between the taps.

Two enlarger and two reducer sections were installed with pipe diameter ratios of 1 to 2 for the enlargers and 2 to 1 for the reducers. One enlargement and reduction was made with 45° conic sections. The up-stream pressure tap at the enlargement was at one 6-in. pipe diameter distance from the section. Three pressure taps down stream from the section were at 1, 2, and 3 12-in. pipe diameter distances. The conic reducer

RICHARD E. DURST, Professor of Chemical Engineering, University of Maine, Orono, Me.

* This investigation was aided by TAPPI Research Grant No. 136.

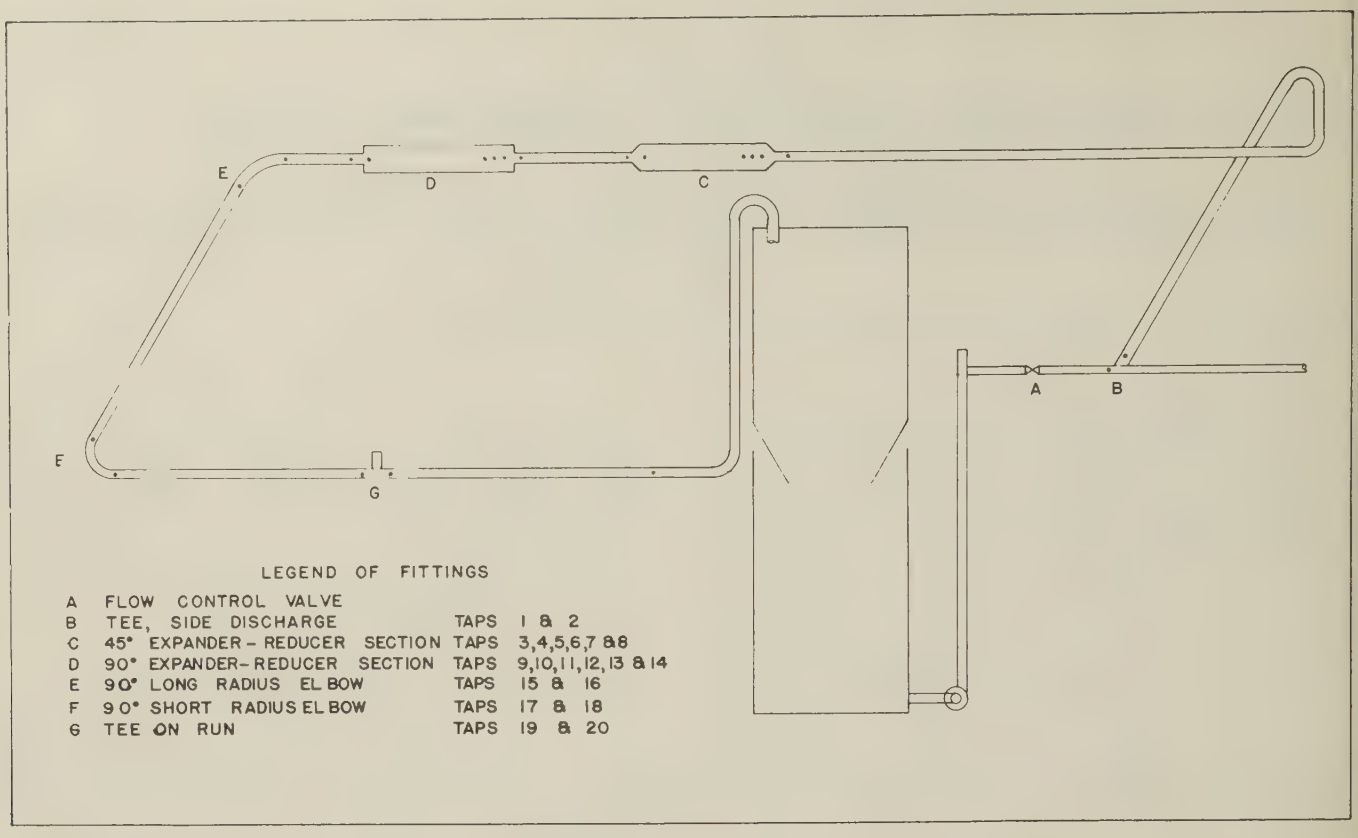
E
D
C
A
B
F
G
LEGEND OF FITTINGS
A FLOW CONTROL VALVE
B TEE, SIDE DISCHARGE TAPS 1 & 2
C 45° EXPANDER - REDUCER SECTION TAPS 3,4,5,6,7 & 8
D 90° EXPANDER - REDUCER SECTION TAPS 9,10,11,12,13 & 14
E 90° LONG RADIUS ELBOW TAPS 15 & 16
F 90° SHORT RADIUS ELBOW TAPS 17 & 18
G TEE ON RUN TAPS 19 & 20

Fig. 1. University of Maine pulp flow system as modified with fittings

had pressure taps 1 pipe diameter each up and down stream from the section. The second enlargement and reduction were 90°. The taps at the enlarger were at 1 pipe diameter upstream and 1, 2, and 3 pipe diameters down stream from the section. The reducer taps were each 1 pipe diameter from the reducer face.

The straight pipe sections between the fittings were so connected into the pressure transmission system that the pressure drop could be taken across each of them as well as across the fittings. The test sections and fittings were designated by the pressure tap numbers as given in Table I.

RESULTS

The pressure drop over the test sections in the flow system was determined for an unbleached northern kraft pulp at 2.0, 3.0, and 3.8% consistencies.

The system was designed on the assumption that a tee on run would have little effect on the straight section following it due to minor deviations of the pressure drop for the tee from the pressure drop for a corresponding straight section of pipe. Test section 20–21, 19 ft. long was considered on this assumption as being a reference section, isolated from the next closest fitting by the tee on run and a 9.7 ft. straight section of pipe to act as a calming section. The equivalent length of the tee on run, as will be shown later justified the assumption. Figure 2 shows the pressure drop curve for the reference section as determined for unbleached kraft pulpstocks at the three consistencies studied. The curves on this plot for 2 and 3% consistencies were compared with the curves obtained on the same stock in long straight sections of the 6.343-in. i.d. pipe in 1954. The new data average about 12% higher than the earlier data. Considering the possibility of alteration of the pipe inside surface during the 3-yr. interval the agreement is quite good.

Each type of fitting will be considered individually. The equivalent lengths in pipe diameters for the short and long radius elbows are shown in Fig. 3. The fittings were approximately 2 and 3 pipe diameters in length, respectively. The equivalent length of the

Table I. Test Sections Descriptions

Pressure tap numbers	*Test section and dimensions*
1–2	Tee, side discharge, 6.343 in. i.d.—length 15 in.
3–4	45° Taper expander, 6.343 to 12.468 in. i.d. taps each 1 p.d. from fitting—length 21 in.
3–5	45° Taper expander, tap 5,2 p.d. from fitting—length 33 in.
3–6	45° Taper expander, tap 6,3 p.d. from fitting—length 45 in.
7–8	45° Taper reducer, 12.468 to 6.343 in i.d. taps each 1 p.d. from fitting—length 21 in.
8–9	Straight pipe section, 6.343 in. i.d.—length 8.33 ft.
9–10	90° Expander, 6.343 to 12.468 in. i.d. taps each 1 p.d. from fitting—length 18 in.
9–11	90° Expander, tap 11, 2 p.d. from fitting—length 30 in.
9–12	90° Expander, tap 12, 3 p.d. from fitting—length 42 in.
13–14	90° Reducer, 12.468 to 6.343 in. i.d. taps each 1 p.d. from fitting—length 18 in.
14–15	Straight pipe section, 6.343 in. i.d.—length 8.85 ft.
15–16	Long radius elbow, segmental welded, 6.343 in. i.d. $R/D = 2$. Taps on ends of fitting—length 18 in.
16–17	Straight pipe section, 6.343 in. i.d.—length 9.7 ft.
17–18	Short radius elbow, segmental welded, 6.343 in. i.d. $R/D = 1.4$. Taps on ends of fitting—length 13 in.
18–19	Straight pipe section, 6.343 in. i.d.—length 9.7 ft.
19–20	Tee on run, 6.343 in. i.d.—length 15 in.
20–21	Straight pipe section, 6.343 in. i.d.—length 19 ft.

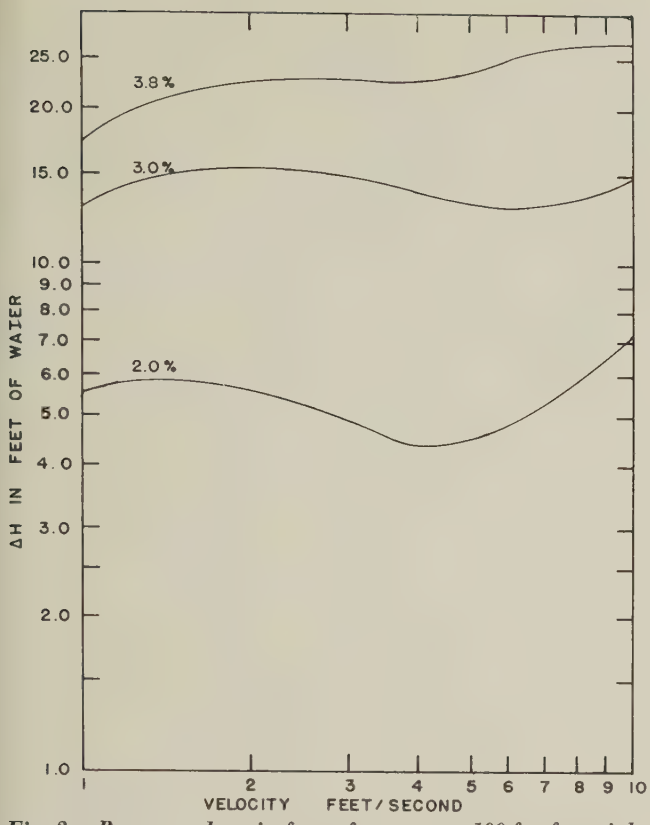

Fig. 2. Pressure drop in feet of water per 100 ft of straight pipe

fitting is clearly seen to decrease with increasing consistency, and to increase with increasing velocity. Minor breaks occur in the curves for the 3.0 and 3.8% consistency stocks at velocities to about 5 and 6 ft. per sec. The breaks will be found to occur in the curves for the other fittings also. The segmental welded elbows, either short or long radius contributes only a small increase in equivalent length to a piping system carrying the heavier pulp stocks.

The results of the study of the pressure drop across tees are shown in Fig. 4. A tee on run can be considered as straight pipe for the consistencies studied. The contribution of the tee side discharge to the total equivalent length of the flow system increases rapidly with velocity for each consistency. The breaks in the curves in these plots occur at the velocity represented by the midpoint between the first maximum and minimum in the normal head loss curve for straight pipe. This would seem to indicate that turbulence is set up in the fitting at lower velocities than in the straight pipe sections, a result that would be anticipated from general knowledge and experience in fluid flow.

The reducers, 90° and 45°, show equivalent lengths decreasing with increasing consistency, Fig. 5, and in each case the 45° reducer produces less effect than the 90° reducer. Again the breaks in the curves occur in the velocity range bracketed by the maximum and minimum for the normal straight pipe head loss curve.

The expanders, either 90° or 45°, resulting in a 1 to 2 increase in pipe diameter do not affect the equivalent length of the system to any appreciable extent. Figure 6 shows the equivalent lengths versus velocity for both types of expander, measuring the pressure drop over the length represented by taps one pipe diameter each from the fitting. The general trend is to exhibit a small pressure recovery with the data falling in a

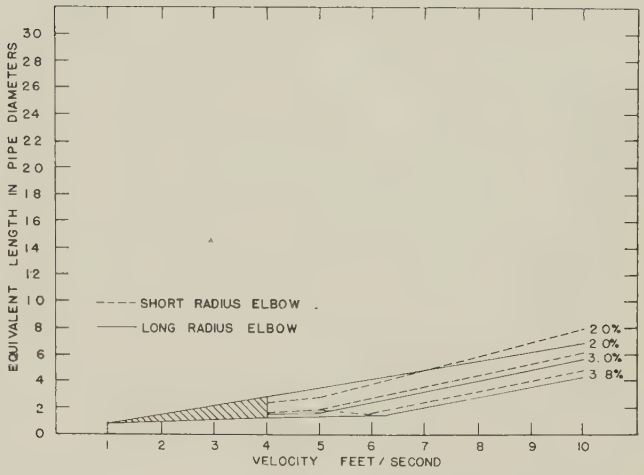

Fig. 3. Equivalent length in pipe diameters for 90° short and long radius elbows at 2.0, 3.0, and 3.8%

narrow band, except for the 2.0% stock in the 45° expander at the lower velocities. Location of the down stream pressure tap at 2 or 3 pipe diameters from the expander did not alter the general pattern except in some few cases to give data points outside the band shown in Fig. 6.

The results of greater significance than the equivalent length of fittings are the effects the fittings have on the straight sections of pipe following them. The effect of the 45° reducer on the straight section 15.75 pipe diameters in length following the reducer is shown in Fig. 7, in which curve 1 is the head loss curve for the reference section calculated to a length equivalent to the test section following the reducer, curve 2. The tendency to reduce the pipe friction due to the presence of the reducer increases with consistency and becomes a considerable factor as the velocity increases.

The 90° reducer produces the greatest effect on the more dilute stocks, Fig. 8. In this plot curve 1 again refers to the reference section calculated to correspond to the length of the test section, 16.72 pipe diameters.

The elbows produce their effects on the straight sections following them as shown in Fig. 9. Here curve 1 is the reference section pressure drop calculated to a length of 18.32 pipe diameters to correspond to the sections following the elbows; curve 2 is for the section following the short radius elbow. The constancy of

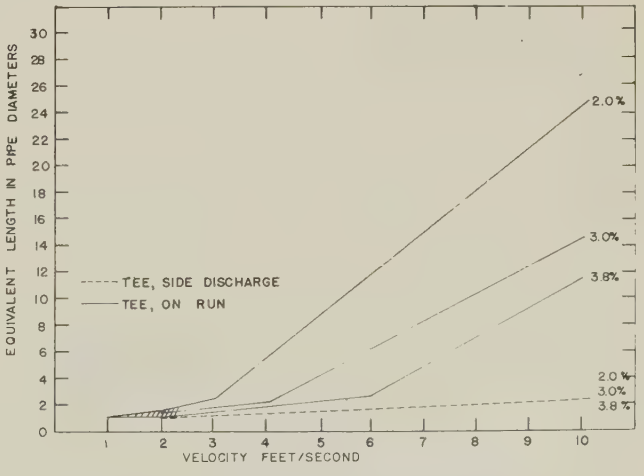

Fig. 4. Equivalent length in pipe diameters for tees side discharge and on run at 2.0, 3.0, and 3.8%

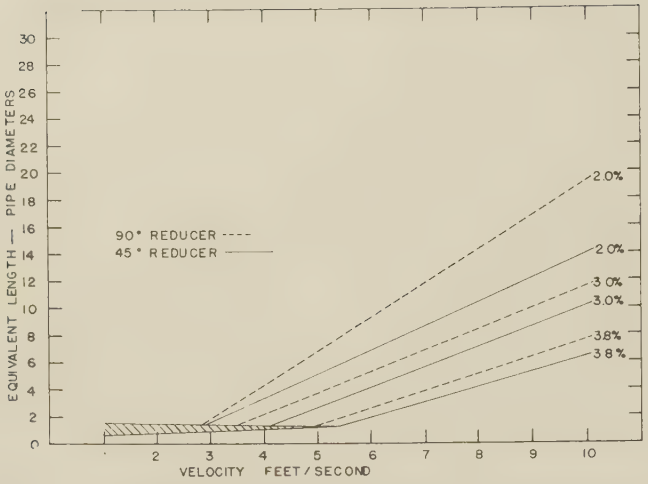

Fig. 5. Equivalent length in pipe diameters for 45° and 90° reducers at 2.0, 3.0, and 3.8%

the effect of increasing velocity to reduce the head loss following fittings is continued for the elbows. The percentage reduction of the head loss in the straight section following the long radius elbow increased from about 8% at 4 ft. per sec. velocity to 35% at 10 ft. for the 3.8% consistency stock. For the short radius elbow the percentage reductions for the same velocity range was 9 to 55%. The 3.0% stock showed reductions of the head loss for the long radius elbow of 4 to 42% and for the short radius elbow of 9 to 71% for the same velocity range. Finally, the 2.0% stock showed almost duplicate curves for the reference section and straight section following the long radius elbow to velocity of 5 ft., at 6 ft. the reduction was 13% increasing to 38% at 10 ft. The straight section following the short radius elbow showed a reduction of head loss of 14% at 4 ft. increasing to 75% at 10 ft.

DISCUSSION OF RESULTS

The flow of pulp stock in pipes has thus far yielded anomalies of such character that no adequate theory has been proposed which will permit the frictional losses to be calculated for other than the simplest conditions. Even such calculations are not without considerable possible error. The correlation (*16*) of pipe friction with shear stress and other physical properties of the pulp and pulp slurries is accurate within about ±20% for straight pipe calculations.

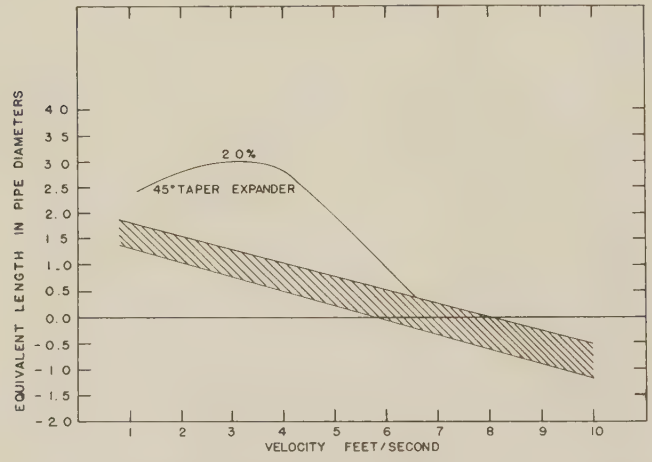

Fig. 6. Equivalent length in pipe diameters of 45° and 90° expanders at 2.0, 3.0, and 3.8%

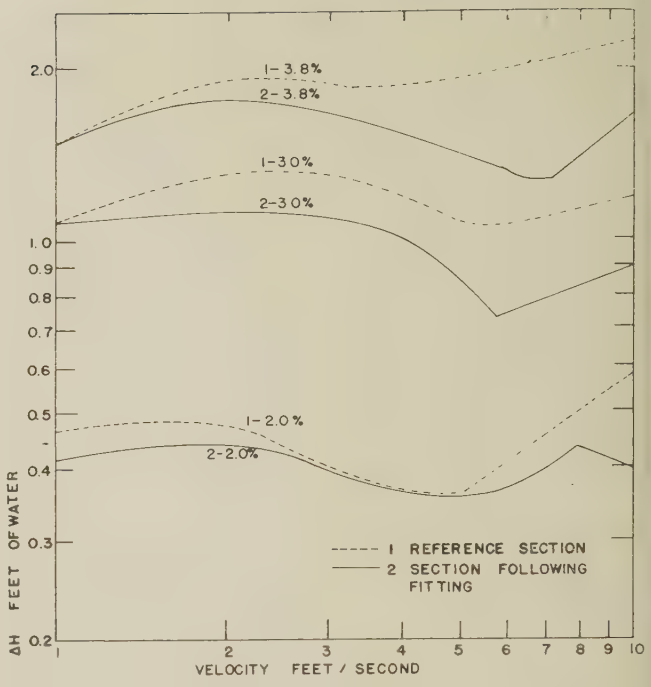

Fig. 7. Effect of 45° reducer on straight section 15.75 p.d. long following the fitting

The results of the study on fittings and straight sections following them up to about 18 pipe diameters in length indicate that flow in such sections behaves in a manner far different than in straight pipe in so-called steady-state flow.

The fittings add to the effective pipe length a number of pipe diameters of equivalent length ranging from a very small value for the tee on run, increasing through the long and short radius elbows, the 45 and 90° re-

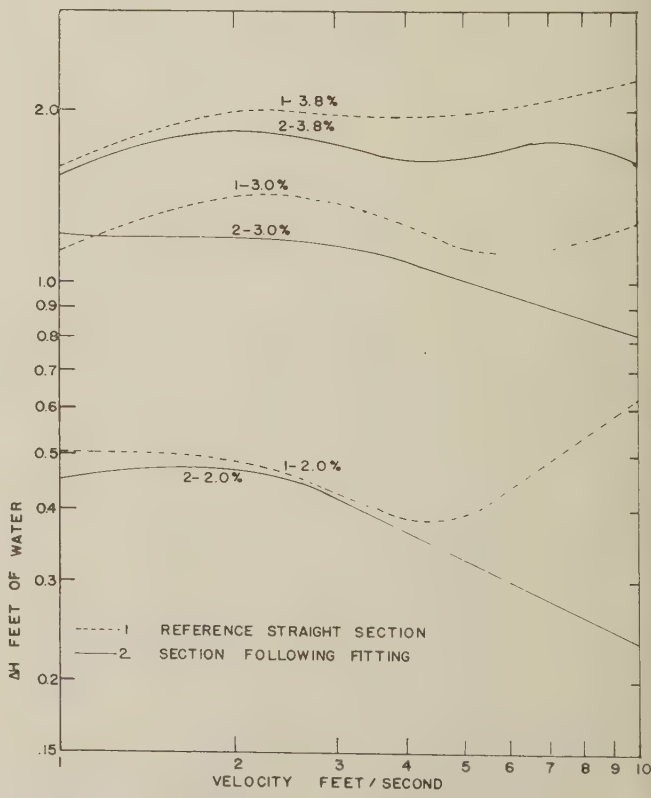

Fig. 8. Effect of 90° reducer on straight section 16.72 p.d. long following the fitting

ducers to a maximum for the tee on side discharge. The reduction in equivalent lengths with increasing consistency can be attributed to the increased calming effect on the flowing stream caused by the larger number of fibers present in the higher consistency stocks. The breaks that occur in these curves indicate the onset of turbulence which appears at increasing velocity with increasing consistency for the same reason the equivalent length is reduced.

The results of greatest significance are those shown in Figs. 7, 8, and 9. The curves in these charts show the straight sections following the reducers and elbows have lower friction head losses than does the straight section of equal length which section is isolated from the effects of fittings by calming lengths. No reference in the literature has been found that would indicate that such

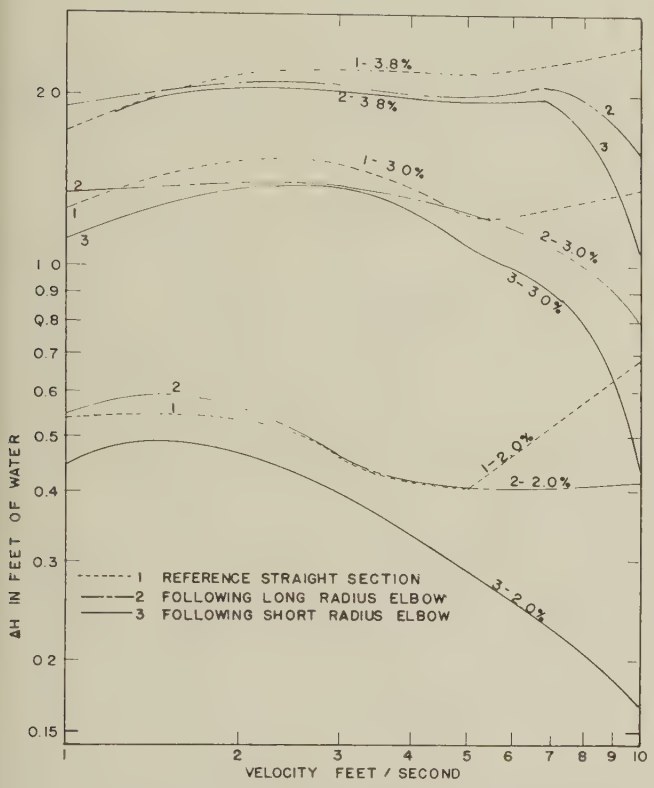

Fig. 9. Effect of long and short radius elbows on straight section 18.32 p.d. long following the fitting

phenomena have been observed by other workers in the fields of fluid or suspension flow. There seems to be no adequate theory that will explain the reduced head loss in the short section of straight pipe following the fittings used in this study. The investigation is being continued at the University of Maine under a program of extended graduate research, developing new and different approaches for analyzing pulp flow patterns.

Some interesting calculations can be made from the data and curves that have been presented. Consider that portion of the flow system from the 90° reducer to the end of the reference test section. The test sections included and for which pressure drop data are available are: 1, straight section 16.72 pipe diameters (p.d.) long following the reducer; 2, long radius elbow; 3, straight section 18.32 p.d. long; 4, short radius elbow; 5, straight section 18.32 p.d. long; 6, tee on run; 7 straight section 35.8 p.d. long (reference section). The total length of pipe and fittings in these sections is 96.4 p.d. or 50.9 feet. For 2% stock at 10 ft. per sec. velocity the equivalent lengths of the elbows and the tee are 7, 8, and 2.5 p.d., respectively. The total length of the system for the given condition is 106.7 p.d. or 56.4 ft. The total of the measured pressure drops is 3.88 ft. of water. From Fig. 2 the pressure drop for 50.9 ft. of pipe in an isolated section is 3.67 ft. of water. A similar study for the same stock at 6 ft. velocity yields 2.64 ft. of water, measured pressure drop for an equivalent length of 99 p.d. or 52.4 ft. of straight pipe, and for the isolated section of 50.9 ft. of length, the pressure drop would be 2.45 feet of water. The 3% stock yields similar analyses at the same velocities. The equivalent length for the test sections is 102.7 p.d. or 54.2 ft. at 10 ft. velocity. The sum of the measured pressure drops is 6.86 ft. of water while the pressure drop for 50.9 ft. of isolated straight pipe would be 7.38 ft. of water. At 6 ft. velocity with an equivalent length of 96.2 p.d. or 50.8 ft. the measured pressure drop is 6.77 ft. of water and for the isolated section of 50.9 ft. it would be 6.62 ft. of water.

These calculated examples would seem to indicate that the pressure drop of the fittings which increases the equivalent length of the system is balanced by the reduced pressure drop of the straight sections following the fittings. The total effect of fittings and straight sections following them is to give an equivalent length of straight pipe which can be considered as having a pressure drop equal to that of an isolated straight section of measured pipe length. Until further studies substantiate the proposition it should be used with the benefit of practical experience.

CONCLUSIONS

The current study of pipe friction for pulp stocks has added to the factual knowledge of pulp flow. It has not yielded a theoretical basis for explaining the anomalies observed.

Specifically, it can be concluded that only the tee on side discharge and reducers add significant equivalent lengths to piping systems, and only at the higher velocities of flow for consistencies of 2.0% and lower.

The straight sections of pipe up to about 20 pipe diameters following fittings have pressure drop values at significantly lower values than do straight sections isolated from fittings by calming lengths. The reduction in pressure drop in these sections becomes greater at the higher velocities of flow.

This study points out the need for more investigation into the behaviour of fibrous suspensions flowing in pipes and fittings.

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Effect of Speed on the Precision of the Tensile Test of Paper

T. W. LASHOF, C. P. SPRING, and G. L. MATON

A round robin was conducted to determine whether any loss in precision would result using a fast test (5 to 9 sec. to break) rather than a proposed slower test (17 to 25 sec. to break) to determine the tensile strength of paper. The study was made using 40, 50, and 60-lb. multiwall kraft paper, both in the machine and cross directions. Three makes of instruments with three laboratories per instrument were involved. It is concluded that speed within the range studied has negligible effect on both the within and between-laboratory variabilities for this type of paper. Expected higher tensile strengths were obtained with the faster test. A by-product of this study is the conclusion that for shipping sack papers five replications are sufficient for the tensile strength test.

While not excluding other types of testers, the existing TAPPI Standard T 404-m-50, Tensile Breaking Strength of Paper and Paperboard, was written primarily for pendulum-type testers. In attempting to broaden the applicability of the method, the TAPPI Paper Testing Committee considered a uniform "time to break" requirement for all papers instead of the present procedure which varies with the strength of the paper and includes a "constant rate of loading" requirement. The proposal met with favor in the TAPPI Paper Shipping Sack Testing Committee, which also uses this standard, but the latter committee recommended a faster test. Consequently the TAPPI Precision Committee was asked to help set up a round robin test to determine whether speed would have an appreciable effect on the between and within-laboratory precisions of the test. The study reported herein was therefore sponsored by the three above-mentioned committees.

The proposed time-to-break requirement was that "the rate of operation of the tester shall be set so that the test will be completed in 20 ± 10 sec., except that for precise work the test shall be completed in 20 ± 3 sec." The Paper Shipping Sack Committee recommended that a time to break of less than 10 sec. be considered. A paper by Capott, Newman, and Mandel (*1*), which was reviewed in manuscript, presented evidence that a time to break of less than about 4 sec. might give falsely low values of tensile strength with an "inertialess" tester, because the recorder could not follow the strain gage at such a high speed. This information was directly supported by the results of a round robin conducted by the Paper Shipping Sack Manufacturers' Association (PSSMA) (*2*). Because of these considerations and limitations of instrument adjustment, the following two test procedures were selected: 5 to 9 sec. time to break and 17 to 25 sec. time to break.

In order to determine whether the effect of speed depends on the material, as many different classes of materials as possible should be studied. However, the immediate interest centered on shipping sack papers, and since members of the Paper Shipping Sack Testing Committee were to do the work, the study was restricted to the most widely used weights of shipping sack papers (40, 50, and 60 lb.) and two directions of test (machine and cross), making in effect a total of six materials.

The next question in the design of the interlaboratory study was the decision as to what types of instruments should be included. The data in the references previously cited indicated that when the same time to break was used, results obtained by different types of testers are essentially identical, both as to average values and as to precision. However, the data of Capott, Newman, and Mandel while covering several materials, were for only one laboratory, while the PSSMA results while applying to several laboratories, included only one material (but in machine and cross directions). It was decided that both the inertialess and the pendulum-type testers should be included in the study, with several laboratories using each. The final design included three makes of testers: the Instron inertialess tester, the Thwing-Albert pendulum tester, and the Testing Machines, Inc., pendulum tester. Each make of tester was used by three laboratories.

The final step in the design was the decision as to how the specimens were to be cut from the rolls and distributed to the laboratories. Since the emphasis of the study was on the comparison of the reliabilities of results from different times to break, the design shown in Fig. 1 was chosen. This design minimizes sampling variability among specimens to be tested on the same instrument but at the two different speeds.

Two methods of analysis of the data were attempted. In the first, each material was analyzed separately using a "nested" (hierarchical) model, which is shown

T. W. Lashof, Physicist, Paper Section, National Bureau of Standard; C. P. Spring, Quality Control Director, Owens-Illinois Mill Div.; G. L. Maton, Director, Research and Statistics, John I. Thompson and Co.

Table I. Analysis of Variance, Hierarchical Model

Source	DF	Mean squares (theoretical)	Components	Materials, lb. Cross 40	Cross 50	Cross 60	Machine 40	Machine 50	Machine 60
Instrument type	2	$63\sigma_I^2 + 14\sigma_L^2 + \sigma_E^2$	σ_I^2	0	0	0	0	0	0
Time to break	1	$126\sigma_T^2 + 14\sigma_{LT}^2 + \sigma_E^2$	σ_T^2	0.07	0.11	0.08	0	0.24	0.43
Instrument × time	2	$63\sigma_{IT}^2 + 14\sigma_{LT}^2 + \sigma_E^2$	σ_{IT}^2	0	0	0.15	0	0	0
Labs within instrument	6	$14\sigma_L^2 + \sigma_E^2$	σ_L^2	0.29[a]	0.82[a]	0.42[a]	0.80[a]	0.86[a]	0.47
Labs × time within instrument	6	$14\sigma_{LT}^2 + \sigma_E^2$	σ_{LT}^2	0	0	0	0.40	0	0
Error	108	σ_E^2	σ_E^2	0.72	1.63	2.55	1.70	3.35	4.67

[a] The mean squares from which these components were derived were significant at the 1% level. The mean squares for all other nonvanishing components were significant at the 5% level.

in Table I. It will be noted that the time-to-break component of variance was found to be small but statistically significant for five of the six materials. This was expected as it is well known that the measured value of the tensile strength of paper increases with the speed of the test (*3*). The effect of instrument type appears to be completely negligible, while the laboratory-to-laboratory difference even for the same type of instrument is statistically significant. However, the design (Fig. 1) of the round robin was such that laboratory differences could be confounded with sampling variability along the roll. Therefore, a separate study was made in one laboratory after the completion of the round robin study, from which it was found that sampling variability along the roll (between sets) was not appreciably greater than sampling variability within a set for four of the six materials. For the other two materials, the 40 and 50-lb. papers in the machine direction, the sampling variability might be large enough to explain observed differences between laboratories.

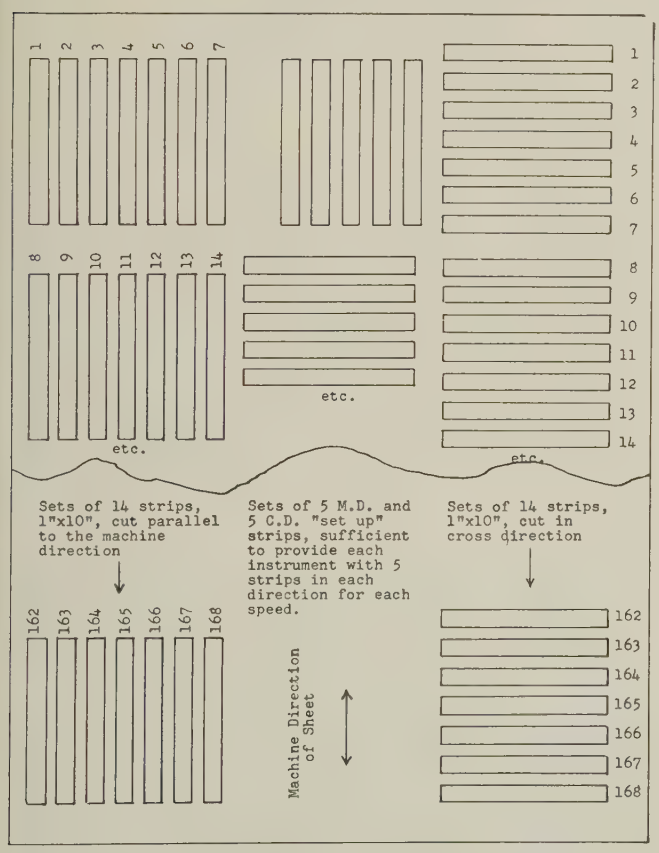
1 2 3 4 5 6 7
8 9 10 11 12 13 14
etc.
etc.
etc.
1 2 3 4 5 6 7
8 9 10 11 12 13 14
Sets of 14 strips, 1"x10", cut parallel to the machine direction
Sets of 5 M.D. and 5 C.D. "set up" strips, sufficient to provide each instrument with 5 strips in each direction for each speed.
Sets of 14 strips, 1"x10", cut in cross direction
162 163 164 165 166 167 168
Machine Direction of Sheet
162 163 164 165 166 167 168

Fig. 1. Pattern for cutting strips for round robin. The pattern shown was followed in preparing specimens for each of the three shipping sack papers: 40, 50, and 60-lb. multiwall kraft papers. A set of 14 consecutive strips was assigned to one instrument, the 14 strips being assigned seven to each speed at random

The second analysis was made in accordance with the "linear model" (*4*), which assumes that systematic differences exist between sets of measurements made by different laboratories, and that these systematic differences are linear functions of the magnitude of the measurements. It follows from this assumption that if the results of each laboratory are plotted against the average results of all laboratories, each laboratory may be represented by a straight line. In the linear model each laboratory is characterized by the slope β and the location parameter μ of its straight line, and the departure η of each experimental point from the line. The ordinates of the experimental points are the average values for each material, the laboratory averages in the case of each tensile study reported here being computed from the seven replications for each material. The abscissas are the average values for all laboratories. The two times to break are treated as separate studies. For both times to break the slopes of the laboratory lines were found to be essentially the same for all laboratories. For the 5 to 9 sec. time to break there also appear no constant differences between the laboratories (variance μ vanishes). For the slower test, 17 to 25 sec. time to break, there do

Table II. Relative Importance of the Various Sources of Variability, Linear Model

No. of replications	Time to break, sec.	Source of variability, % λ	ϵ	Systematic
10	5–9	76	24	0
	17–25	75.5	16	8.5
5	5–9	61	39	0
	17–25	65	28	7
1	5–9	24	76	0
	17–25	30.5	66	3.5

appear some constant differences between the laboratories; however, as will be seen below, this is quite negligible as compared with non-systematic differences between laboratories.

The departure η of an experimental point from its line may be divided into two parts: that explicable by replication error, ϵ, and that which is not, λ. The latter may be thought of as the differential response of that laboratory either to some interfering property of the material (such as response to relative humidity) or as a result of instrument scale error. The effect of ϵ may be reduced by replication but the effect of λ may be reduced only by a careful study and revision of the method. Table II shows the percentage of the total variance which is due to each source of variability when 10 replications are run in accordance with the present TAPPI Standard T 404 m-50 and also when only one and five replications are run. It is seen that even five replications are more than enough to make the variance associated with ϵ smaller than that associated

Table III. Comparison of Tensile Breaking Strength and Precision of Test for Two Speeds of Test

Material	Fast, 5–9 sec. Av., lb.	Fast, 5–9 sec. Std. dev., lb.	Slow, 17–25 sec. Av., lb.	Slow, 17–25 sec. Std. dev., lb.
40-lb. cross	16.1	1.1	15.5	1.2
50-lb. cross	19.4	1.4	18.7	1.4
60-lb. cross	25.0	1.8	24.4	1.6
40-lb. machine	26.1	1.9	25.4	1.7
50-lb. machine	32.3	2.3	31.3	2.0
60-lb. machine	39.3	2.8	37.9	2.3

with λ. (Three replications would be enough except that the analysis is somewhat uncertain because of the confounding with sampling variability previously mentioned.) Hence for the shipping sack papers included in this round robin, it appears that it would be wasteful to base a test result for tensile strength on more than about five specimens.

Table III shows the average values and total standard deviations (one replication) for each material involved in the round robin. The total standard deviation includes the effect of all three sources of variability listed in Table II. It will be noted that there is little difference between the total standard deviations for the two speeds. Therefore it can be concluded that with regard to precision, the procedures are equivalent. Also since the two procedures yield nearly the same values of tensile strength, their sensitivities (*5*) are equivalent. The differences in tensile strength obtained by the two procedures are in the expected direction and of the expected order of magnitude (*3*).

In view of the above experimental results, it must be concluded that the 5 to 9 sec. time to break yields equivalent results to those obtained by the 17 to 25 sec. time to break, and that therefore the 5 to 9 sec. time to break, because it is faster and requires less testing time, is the better procedure for determining the tensile strength of shipping sack papers.

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Animal-Glue Testing for Paper Sizing

I. Test Methods Investigated

P. B. DAVIDSON and H. B. BODENHAGEN

A standard laboratory tub sizing and air-drying procedure was developed that possessed reasonably good reliability and duplicated, as far as possible, the conditions that obtained in the mill processes. A numerical measure of penetration, based upon the determination of reflectance, was perfected which gave a statistical evaluation of sizing efficiency. Various testing methods were either adapted to or developed for determining glue characteristics that might be pertinent to sizing efficiency. Some of these methods were found wanting and were discarded while others received further attention for the purpose of increasing their precision and reliability. A preliminary program was set up to test a large number of both hide and bone glues by the full list of test methods. From the results of this program nine representative hide glues were selected for more careful testing at both pH 7 and pH 9 by the refined methods. The results of these tests verified the supposition that gel strength and viscosity did not adequately define the properties of a glue for tub sizing of technical papers. The time required for formaldehyde to react with glue at a pH of 9 was very useful in differentiating bone from hide glues. The change in viscosity of a glue between pH 7 and pH 9 gave some indication to the class of glue which could be used for blending to give a specified gel strength and viscosity. The amount of water required to bring the viscosity of a 7.7% glue at 45°C. to the same viscosity at 25°C. was, in general, a good index of sizing efficiency. A sensitive surface-tension test, made with the Instron tester, not only produced a charted record of the surface tensions of the various glues but also gave a measure of the stretchability or toughness of the surface film on the glue. The full significance of this film elongation has probably not been realized, but this new measurement indicated the deleterious effects of zinc compounds, used as preservatives, upon glue. It is still uncertain whether the chromatographic method detects small concentrations of disintegration products in both hide and bone glues or just of bone glues. This much appeared evident, however: when such products were found to be present, the sizing efficiency of that particular glue was poor.

THE surface sizing of paper with animal glue is one of the oldest processes used in papermaking. When Illig revolutionized paper sizing with his invention of rosin size, *ca.* 1807, surface sizing became more of a supplementary operation that not only increased the degree of sizing but produced special surface and strength properties in the paper. Such specialized properties as wet tensile strength, resistance to wet abrasion, and high resistance to liquid penetration have become particularly important in present-day photosensitive and other technical papers. There was a time, well within the memory of the present generation of papermakers, when paper mills prepared their own animal glue by cooking hide clippings. The mills abandoned this practice in the face of increasingly stringent demands

P. B. DAVIDSON and H. B. BODENHAGEN, Mellon Institute, Pittsburgh, Pa.

upon technical paper properties, and found it necessary to purchase better grades of glue on specification.

With minor consideration given to grease content and foaming tendencies, the time-honored specifications for animal glue have been gel strength and viscosity and it has been common practice for glue manufacturers to blend various grades of glue to obtain the particular gram strength and viscosity the papermaker thought he needed and could afford to use. But to the papermaker faced with narrowing tolerances in critical properties of his technical papers there has come the realization that gram strength and viscosity do not serve as adequate criteria for the properties or use requirements of his animal-glue tub sizing.

The raw materials from which hide glue is extracted are hide clippings and fleshings from tanneries. Bone glue is obtained from both green and old bones. A brief review of the process by which glue is made from such stock clearly shows why gram strength and viscosity are inadequate specifications for a specialized use for glue. In the first place, unless proper precautions are taken, bacterial degradation of the protein material in the raw stock can easily take place during its storage. Further problems occur during manufacture.

The extraction process for hide glue starts with the soaking and swelling of the hide clippings in milk of lime for a period of several weeks. The limed hides are then thoroughly washed to remove solubilized albuminous and mucinous material as well as lime and its hydrolysis products. The residual alkali left in the swelled hides is neutralized with either hydrochloric, sulphuric, phosphoric, or sulphurous acid, and washing is continued to decrease the salt content to a minimum. Glue is extracted from these swelled hide clippings by water cooking them in vats heated by steam coils to about 140°F. The bottoms of these vats are generally covered with a layer of excelsior or other filtering material. The extraction, or "boiling" process, is essentially one of hydrolysis plus solution and thus is a time-temperature concentration reaction. At the end of the cooking period the dispersed glue is drawn off through a valve in the bottom of the vat. The vat, containing the partially extracted clippings, is refilled with water and a second cooking made at a temperature slightly above that of the first cook. Four or more such cooks are made, the last one being at a temperature of about 212°F. Filtration of each batch of extracted glue is necessary to remove suspended matter such as undissolved organic matter, albumins and mucins, lime soap, grease, hair, and mineral particles. From this point on the chief process involved is the removal of water or the concentration of the glue to the point where it can be chilled to a gel and dried to a solid containing about 12% moisture.

This highly simplified description gives the essentials of the process but does not include the addition of bleaching agents, preservatives, and defoamers that may or may not be desired. Throughout the whole process the high-molecular-weight protein material is subjected to certain amounts of unavoidable bacterial and hydrolytic action that result in disintegration products of unknown identity. In none of the draws of glue liquor from the cooker is the liquor completely removed and the glue solids in the retained liquor are further hydrolyzed during the next cook. As a result of this hydrolysis, the derived proteins that constitute glue are degraded beyond the limitations of good quality. For many purposes the blending of glues from different cooks produces a satisfactory product provided its viscosity and gel strength are within specified limits. Such limits define the grade of the glue. It is recognized, however, that "while this system of grading serves a good purpose in establishing a relatively simple basis for evaluating the product and provides a rough guide for glue use in most applications, it should be noted that such tests do not always accurately measure their working qualities" ("Animal Glue in Industry," published by the National Association of Glue Manufacturers, Inc.).

It was with this inadequacy in mind that the authors launched upon a study of what glue properties were required for tub sizing of technical papers and the development, if possible, of test methods that would serve for the proper evaluation of animal glue. The major paper properties to be considered were (1) sizing efficiency, (2) wet-strength, and (3) coating, i.e., photosensitive coatings with blueprint, diazo, and negative solutions.

Laboratory Sizing Method

Before any glue-testing method could be evaluated against use requirements a reliable laboratory tub-sizing process was required; one that would not only tub size paper samples under controlled conditions but that would duplicate, with reasonable accuracy, the mill tub-sizing process. A size press had been built utilizing both a hard-rubber wringer roll and an aluminum roll of 2 5/8-in. diam. The rolls turned on ball bearings, the housings of which rested in slots cut in the upright wooden supports (Fig. 1). A flat aluminum pan was made for placement in a large pan that served as a water bath. These pans were placed on a hot plate and at such a height that the rubber, or bottom, roll dipped into the glue size contained by the smaller of the two pans. The bottom roll was hand operated with a crank threaded into the end of the roll shaft. When the size, of 7.7% concentration, had been brought to the proper temperature of 45°C., strips of paper 14 by 4 in. were immersed in the size and any air bubbles formed on the surface brushed off with a bent glass rod. Excess glue was squeezed off by passing the samples through the nip of the rolls under a total roll pressure of 160 lb. The stack of eight or ten sized-paper samples was allowed to

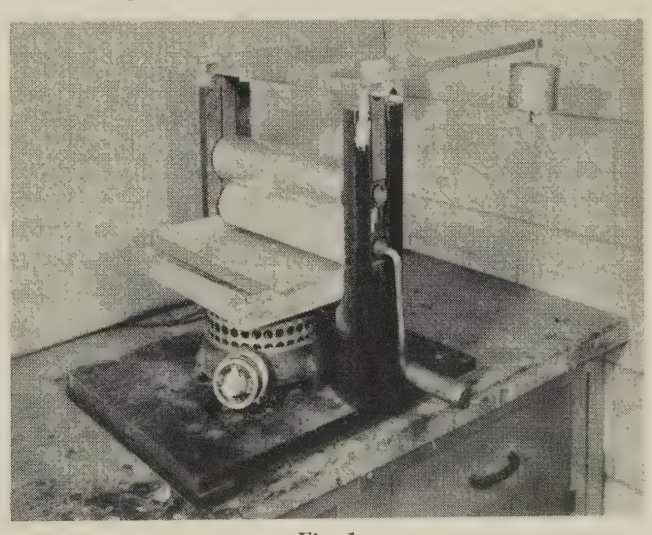


Fig. 1.

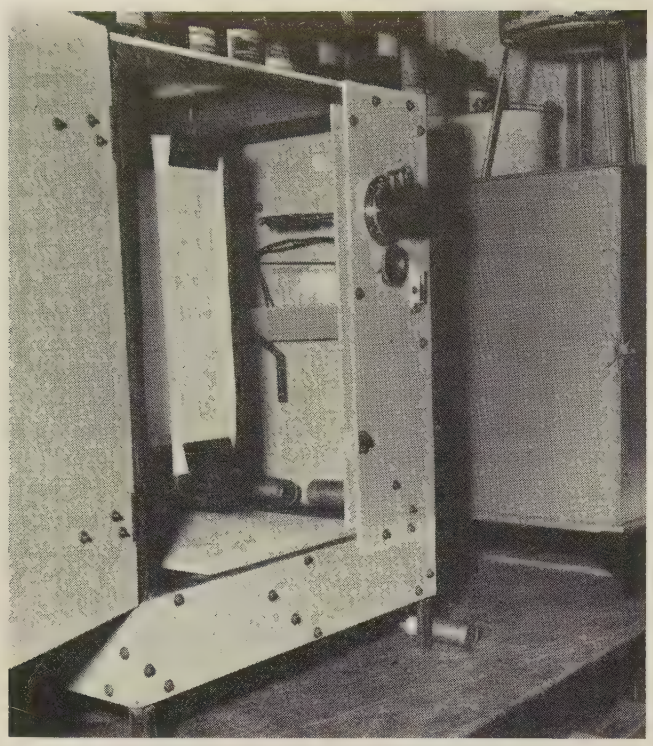


Fig. 2.

"cure" for 30 min. between cool, moist papermakers' felts, after which each sample was treated with a 2% formaldehyde solution in the size press and dried.

Drying consisted of two operations: the first was air drying in a stream of hot air and the other, final drum drying in a Pako Electro-gloss photo drier. For the air-drying process, a cabinet was built of Transite (Fig. 2) and equipped with a small double centrifugal blower, bimetal thermoregulator, a dial-type thermometer, two sets of screw-base cartridge heating elements (two 150 and two 100 w.), and an electronic relay. Baffles were placed in the cabinet to direct the flow of air as it was blown past the heaters and back onto the paper samples. One set of heaters was used to bring the air close to the required temperature and the other to keep the temperature regulated to within $\pm 0.5°$F. The cabinet was heat-insulated with batts of glass wool held in place behind aluminum foil. Large paper clips were placed at both ends of each sized paper sample and five or six samples were strung onto a small steel rod which passed through holes in the clips. The rod carrying the samples was then placed on hooks in the top of the cabinet and drying of the samples started. A time-temperature drying curve was established by drying sets of sized papers at temperatures ranging from 54 to 88°C. At 1-min. intervals a sample was quickly removed from the rod in the cabinet and its moisture content determined. From these drying data a selection was available of either a fast-or slow-drying schedule that would bring the moisture to between 10 and 15%. The final drying to about 4 or 5% moisture was accomplished by adjusting the Pako drum drier to its fastest speed and a temperature of 121°C. These approximate conditions were obtained with 1-min. cabinet drying at 88°C., 2 min. at 77°C., 3 min. at 66°C., and 4 min. at 54°C.

Measure of Tub-Sizing Efficiency

The basic requirement for a sizing efficiency test was that it should be realistic; i.e., if a blueprint paper sizing was being considered, the test should measure penetration of blueprint coating solution and not diazo solution or a fountain-pen ink. Previous experience with penetration tests for rosin or engine sizing had well illustrated this requirement and the blueprint coating solution selected for penetration tests was one known to be representative of those used in the industry. Experience had also taught that visual observation of the amount of penetration of the coating solution was too subject to variations in human judgment to be infallible. Some method was required that would provide a numerical measure of penetration rather than a classification of papers as good, fair, and poor from visual examination. The obvious procedure was that of making reflectance measurements on the uncoated side of developed blueprints. The Densichron reflectance meter was selected for this purpose, but it was found that some minor changes were necessary to obtain differences in reflectance when the beam of light impinged upon a small penetrated area. The small dark area was so overbalanced by the surrounding white area being illuminated that very small differences in reflectance were not being indicated. The regular lens in the Densichron reflectance head was replaced with a different "small-beam" lens that concentrated the light into a beam about half the diameter of that from the original lens. But this beam of light was still too large, so the incident beam was reduced still further to $^1/_{16}$ in. by inserting a vulcanized fiber plug in the light opening of the reflection head. This plug had an accurately centered $^1/_{16}$-in. hole drilled parallel to the axis of the plug and the exposed end, shaped to the contour of the side wall of the reflection chamber. With such a small beam of light incident to the sample, larger differences in reflectance readings were obtained when a dark penetration spot was encountered in the scanning process.

In making a determination the following steps were pursued:

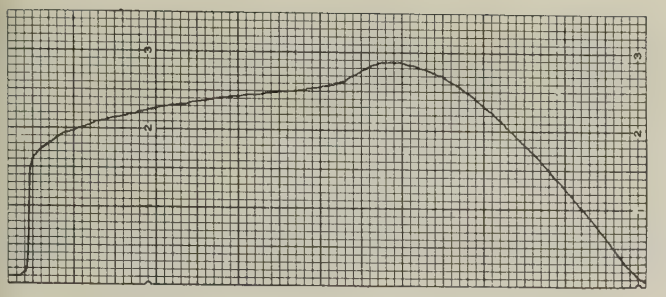
1. The instrument was adjusted to 100% reflectance with the white glass standard and to zero reflectance with the black glass standard.
2. The blueprint, printed and developed on both sides, was placed coated side down against a standard black-glass background. The coatings were applied to sized papers that had been conditioned at 70° F. and 65% R.H. for 24 hr.
3. The light reflected from the sample was passed through a red filter to give greater contrast between the dark blue colored penetration spots and the surrounding white paper.
4. About 80 readings were taken, a number which gave a normal distribution curve.

It was found that variance in reflectance served as a better gage of penetration or sizing efficiency than the mean of the reflectance readings. Variance was calculated from the formula,

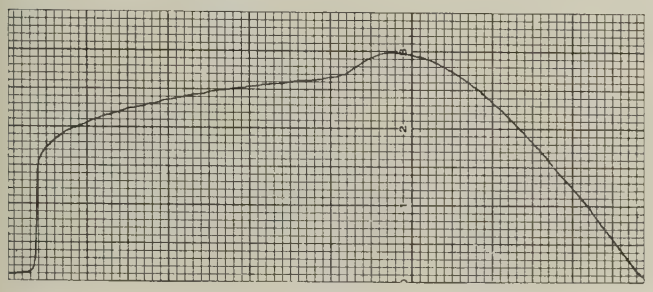
$$\sigma^2 = \frac{\Sigma(x^2) - [(\Sigma x)^2/n]}{n - 1}$$

where σ^2 is variance, x the reflectance reading values, and n the number of readings taken.

Reflectance measurements were made on a number of mill-sized papers, some of which had been rejected owing to poor sizing, and others accepted for shipment.



At pH 6.98 and 46°C.



At pH 9.03 and 46°C.

Fig. 3. Glue Sample A

These papers varied in weight from 24-lb. blueprint to 14-lb. negative paper. Analysis of the data showed that variance limits could be established for either acceptability or rejection for each weight of paper as follows:

24-lb. paper—variance <10, acceptable
20½-lb. paper—variance <10, acceptable
17-lb. paper—variance <20, acceptable
14-lb. paper—variance <25, acceptable

Owing to the amount of permissible "show through" of color in the lightweight 14-lb. papers, the mean of the reflectance values also had to be considered. A mean reflectance of 50 was judged as probably acceptable provided the variance was <25.

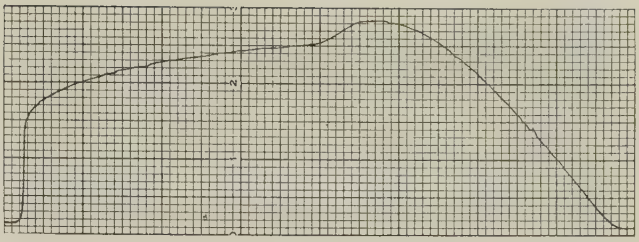
Wet-Strength Test

The wet tensile strength is of primary concern in technical papers because of the tension under which paper is placed either when being coated with the sensitizing solution or developed and washed. For this determination, ½-in. width strips were soaked for 30 min. in water at 21.1°C. and the tensile strength determined in the normal way in an Amthor tester. The results were reported in grams.

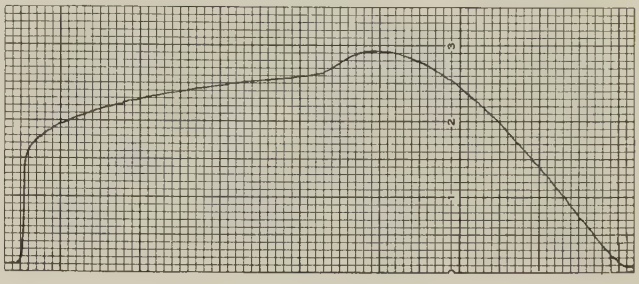
Coating Properties

Two factors were involved in judging coating properties of blueprint paper: color development in the print and complete coverage of the paper by the coating solution. Previous studies of this latter characteristic had shown that the paper was not always completely wetted and covered by the animal glue and, when subsequently coated with blueprint solution, these same small unsized areas were not covered by photosensitive chemicals. This resulted in what is termed white spotting. Tests indicated that this was not necessarily caused by grease in the glue.

As in the case of penetration, some numerical gage of white spotting was required and the same reflectance measurement was used to determine this property. As white spots are usually much smaller than penetration spots, more background was included in the illuminated areas and lower values of variance were obtained. But



At pH 6.98 and 46°C.



At pH 9 and 46°C.

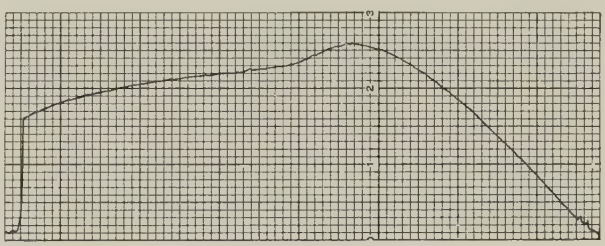
Fig. 4. Glue Sample B

there appeared to be sufficient difference in variance between white-spotted and nonwhite-spotted papers to use the method for judging white spotting. Variance, however, was found in some papers that exhibited no white spots. This variance was caused by dark blue spots that appeared in partially penetrated areas of the blueprints. Such a condition must be kept in mind when this method is used for appraising white spotting. No cases have been observed where both white and dark spotting occurred in the same paper sample.

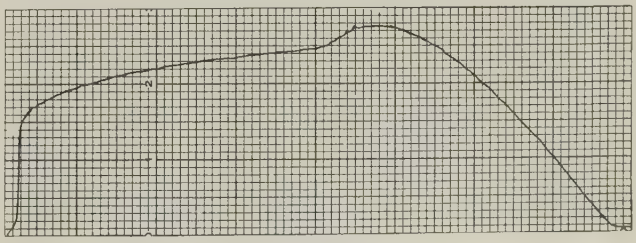
Glue-Testing Methods

A basic assumption had been made that sizing efficiency and strength development of a glue depended upon its gelatin content. Nevertheless, the economics of papermaking pointed to the impracticality of sizing with photographic or other high priced glues. The obvious compromise was to select an adequate "technical gelatin" or good grade of glue, with test methods designed to differentiate the good from the questionable.

The reliability and selectivity of a test had to be determined by applying it to various grades of glue. For this purpose glue samples were obtained from 12 sources.

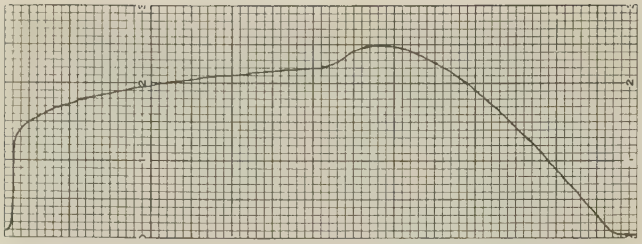


At pH 7.02 and 46°C.

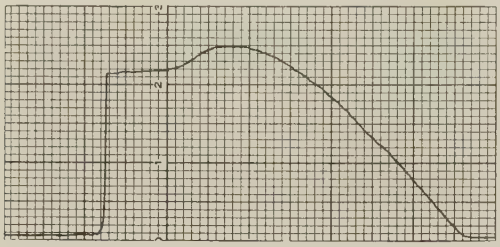


At pH 9.03 and 46°C.

Fig. 5. Glue Sample C



At pH 7.02 and 46°C.



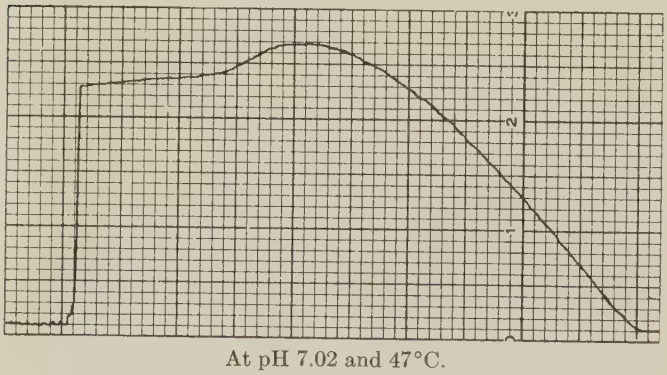
At pH 8.99 and 46°C.

Fig. 6. Glue Sample D

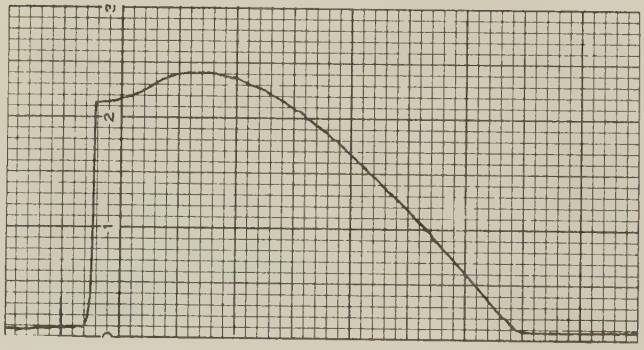
Thirty-six glues were tested, ranging from 600-g.-strength gelatins to 135-g.-strength bone glues.

Owing to the fact that this study was made in an attempt to find new tests for glue, no consideration was given to determining gram strength. Viscosity tests were made on these glues but in a different manner than normally considered by glue manufacturers.

1. *Viscosity.* Rheologists do not regard a flow method as applicable to viscosity determinations of non-Newtonian systems, such as glue and starch dispersions. Theoretically a shearing method, as in the case of the Stormer, MacMichael, or Brookfield instrument, should be used. The official method adopted by glue manufacturers measures the time of flow from a jacketed pipet. Inasmuch as comparative values were adequate for this study and the Ostwald pipet was at least as reliable as the official pipet method, the Ostwald apparatus was used in a constant temperature bath. Viscosity values were reported as relative viscosity, i.e., relative to water at the same temperature. If paper was sized at 45°C. with a 7.7% glue dispersion and the object of the test was to determine use requirements, it appeared logical to find the viscosity of the size under these conditions, assuming that viscosity influences the manner in which the size enters and penetrates the paper. It also appeared logical to calculate the concentration of the size on an oven-dry weight of glue rather than the air-dry weight (as provided in the official methods), particularly in view of the fact that air-dry glue samples vary in moisture content by as much as 4.5 to 5%.

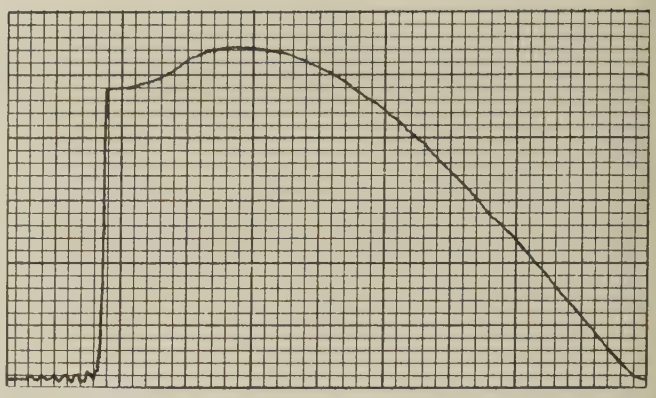


At pH 7.02 and 47°C.

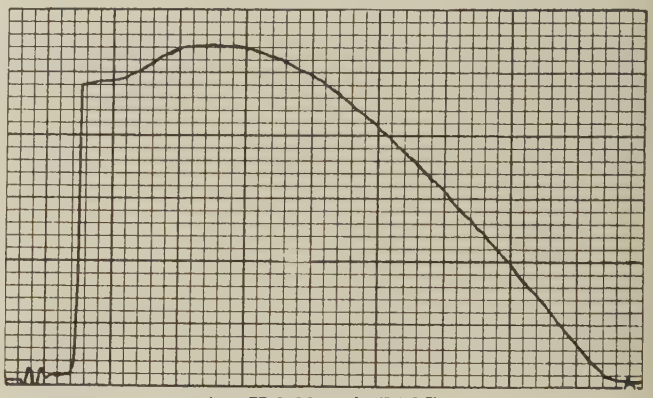


At pH 9.03 and 47.5°C.

Fig. 7. Glue Sample E



At pH 6.99 and 46°C.



At pH 9.00 and 47.5°C.

Fig. 8. Glue Sample F

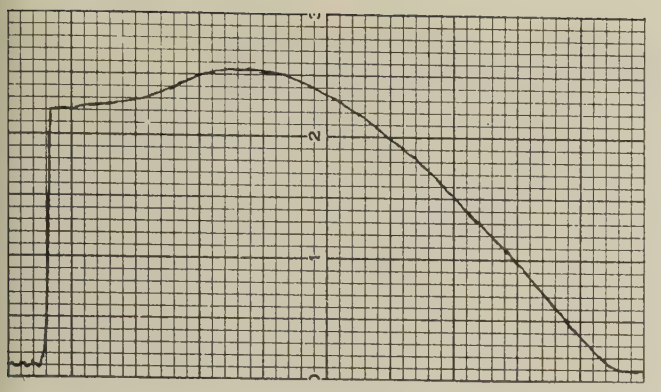
2. *pH Determinations.* The pH determinations were all made with a glass electrode.

3. *Density.* In making relative viscosity determinations the density of the dispersion was a required factor in the following formula:

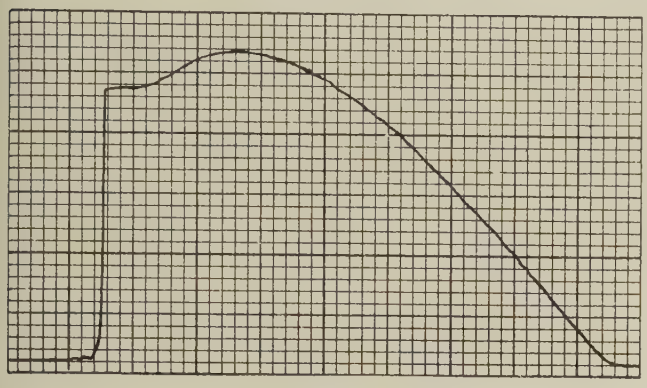
$$\text{Relative viscosity} = \frac{\text{Time of flow of glue} \times \text{density of glue sol}}{\text{Time of flow of water} \times \text{density of water}}$$

The density determinations were made by a pycnometer method.

4. *Softening Point.* One of the difficulties in making a softening point determination—or what is generally termed a melting point—was that of obtaining a sharp end point. The simple time-honored melting-tube method was adapted to measure the softening point of glue gels. Four-inch lengths of 5-mm. glass tubing were sealed off at one end. The tubes were filled about $^3/_4$-full with the glue size and chilled overnight at between 0 and 3.33°C. To determine when the glue started to melt, a small stainless steel rod, cut to $^1/_2$-in.



At pH 7.0 and 47°C.



At pH 9.02 and 47°C.

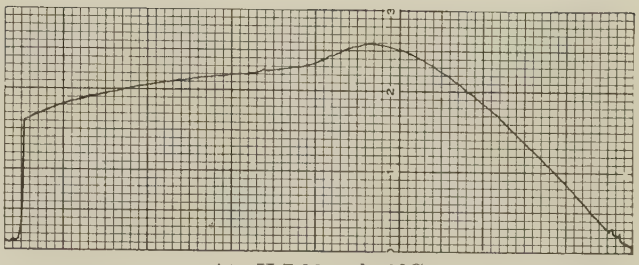
Fig. 9. Glue Sample G

length and filed down to a weight of 0.744 ± 0.0003 g., was inserted in the tube so as to rest on top of the gelled glue. The tube was then attached to a thermometer and placed in a water bath which was slowly heated. The softening point was indicated when the steel rod started to sink. This happened when the layer of glue next to the wall of the glass tube melted and allowed the unmelted plug of glue in the center, with the metal rod riding on top, to move downward. Duplicate determinations made on several glues showed the method to be quite reliable.

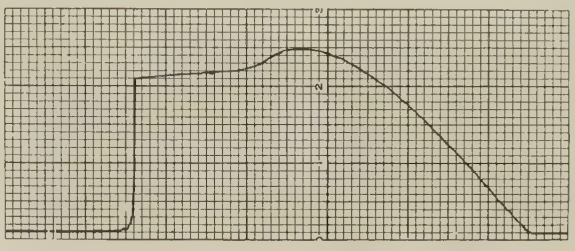
5. *Adhesion Tension.* Adhesion tension is defined as the product of the surface tension of a liquid and the cosine of the angle of contact between the liquid and the solid. The contact angle was measured by a modification of the TAPPI Standard T 458, in which the shadow of a drop of the glue size at the point of contact with the paper was projected through a microscope onto a chart, held in a Euscope, having lines ruled on it to serve as a protractor. The line tangent to the shadow at the point of contact with the paper indicated the angle of contact. No claim is made here that this is a precision method, but it was sufficiently accurate for measuring some of the wide differences in wettability exhibited by the various glues.

In the first glue evaluations, surface tension was determined with the Du Nouy instrument. At a later date it was learned from a private communication that F. T. Seawell of the Corn Products Fellowship and Victor Jackman (*1*) of the Instruments Section at Mellon Institute had developed a more accurate method for surface tension measurements with the use of the Instron tester.

6. *Instron Measurement of Surface Tension.* The same principle was involved in measuring surface tension with the Instron tester as was described by Harkins and Jordan (*2*); that is, both methods measured the force required to pull a platinum ring free from the surface of the liquid system under more carefully controlled conditions than obtain with the Du Nouy instrument. The advantages of the Instron tester were that the application of the force was at a known rate, was constant, and the stress applied was recorded on a chart. This charted record showed not only the surface tension but the elasticity or elongation of the surface film. In agreement with Harkins and Jordan (*2*), the charts showed a maximum point of stress, which was the surface tension, from which the stress curve gradually decreased over varying lengths of time or as the ring was

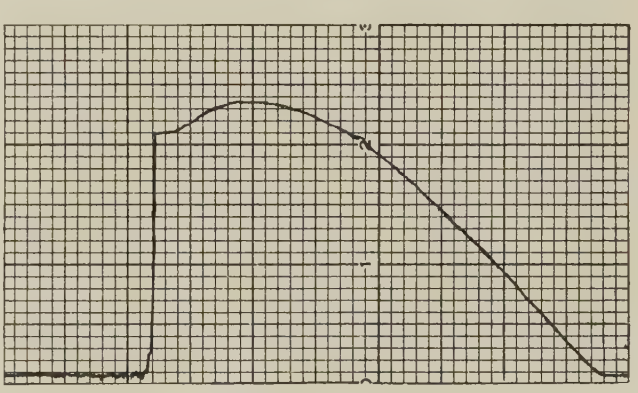


At pH 7.06 and 46°C.

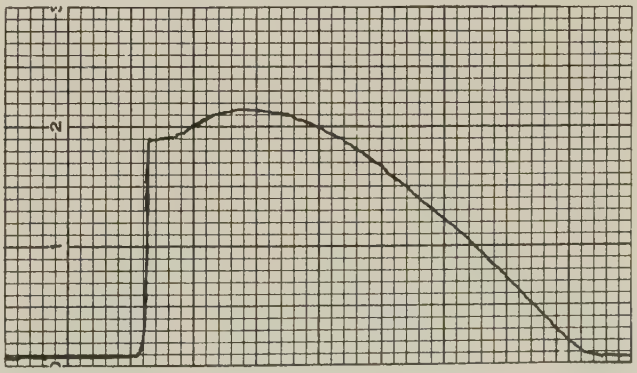


At pH 8.99 and 46.5°C.

Fig. 10. Glue Sample H



At pH 7 and 46° C.



At pH 9 and 46° C.

Fig. 11. Glue Sample I

pulled farther from the surface of the liquid. The varying distance the ring was pulled from the surface before it finally broke free from the liquid indicated varying film-forming properties of the system.

In the first measurements of surface tension of glue dispersions by this method, successive determinations were made on the same samples and it was found that reproducible results were the exception rather than the rule. This was attributed to incipient gelation that probably took place in the glue size. It was also found that a container for the size sample must be large enough to avoid the interfering meniscus formed at the side of the beaker. These difficulties were overcome in Seawell's later refinement of the method, by testing a larger sample, 500 ml. contained in a three-neck round-bottom flask, at a temperature above the transition temperature of the glue. This type of flask allowed an accurate temperature record through use of a thermometer inserted through one neck of the flask and maintenance of temperature by placing the flask in a Glas-Col spherical mantle. By closing off the other small neck of the flask with a stopper high humidity was maintained in the flask, which reduced to a minimum the formation of a partially dried skin on the surface of the glue dispersion. The platinum ring was mounted on a long rod inserted through the large center neck of the flask and attached to the top stationary jaw of the tester. As the bottom carriage of the tester was slowly lowered at a known and constant rate, the immersed ring reached the surface of the glue and strain was recorded on the chart.

7. *Formaldehyde Requirement.* If, in the blending of glues to obtain a specified gel strength and viscosity, some low-grade glues were used that did not react rapidly with formaldehyde, then sizing efficiency and wet-strength development should be affected. In mill operation the time factor is important. Unless the reaction between glue and formaldehyde is rapid some of the formaldehyde is driven off by the heat of the drier before it reacts with the glue. As a method indicating use requirements, some measure of reaction time was desired. A practical end point for such a method would be the loss of fluidity of the glue size, but the problem was how to determine the change from sol to gel state. An approximation method was first used to learn whether such a determination were practical.

Having indicated a distinct value in the appraisal of different blends and types of glues, refinements were made in the method which gave a sharper and more reliable end point. The method consisted of placing 50 ml. of 7.7% glue size in a 100-ml. beaker which, in turn, was placed in a water bath regulated to 45 ± 0.5°C. The size should reach that temperature within 20 min. A steady stream of air was blown through the size by passing it through a glass tube, the tip of which was drawn to an inside diameter of approximately 0.5 mm. The air flowed through at a rate of 55 ml. per min. The formaldehyde solution was added at the instant the timer was started and the timer stopped when the constant stream of air into the size was momentarily interrupted; in other words, when the gel that had formed blocked the steady flow of air. The time recorded was taken as the formaldehyde requirement of the glue. The rate of air flow was determined with a ball-type flow meter, type 316SS float, in a tapered glass tube-range for air 410 to 41 ml. per min.—manufactured by Fischer and Porter Co. At the low rate of air flow through a small tip, the ball in the flow meter remained stationary rather than continually bobbing up and down as when large intermittent bubbles flowed through the tip. At the end point, or gelling of the glue, the flow-meter ball jumped up at the time the steady flow was interrupted giving a possibly better end point than that of observing the momentary interruption of the stream of air in the sample.

8. *R_f Factor.* It is common knowledge that the glue manufacturer blends various grades of glue to obtain a sizing glue having certain specified gel strength and viscosity. There is nothing improper about this; in fact by so doing the glue manufacturer gives the papermaker what he thinks he wants. On the other hand, certain grades of glue lack uniformity from one shipment to another and a quality-control laboratory naturally questions whether the glue-making process has been out of control or whether a different blend of glue has been received. In either case the possible presence of disintegration or hydrolytic products that would lower sizing efficiency made it advisable to find a method for their determination.

Disintegration products were detected in some glues by a chromatographic method described in its essentials in *Science* (*3*). The method was modified by using larger strips of paper (7 in. long, 1 in. wide at one end and $^1/_2$ in. wide at the other) and placing them in 8-in. test tubes so that the bottom of the strip was immersed for about $^1/_8$ in. in the solvent and not touching the sides of the tube except at the wide top of the strip.

A small drop of glue size of 7.7% concentration was placed on the small end of the paper strip of Whatman no. 1 chromatographic paper at about $^5/_8$ to $^3/_4$ in. from the bottom and the drop of glue allowed to air dry. The solvent, 0.8 ml., was introduced into the bottom of the 8-in. test tube. The solvent was composed of 80% ethanol, 5% ammonia (28% NH_4OH), and 15% water. With tweezers, the paper strip was inserted in the test tube so that the small end was immersed in the solvent to a depth of about $^1/_8$ in. Care was taken that the drop of dried glue did not come in contact with the solvent and that fingers did not touch the lower or small area of the paper, thus leaving prints that might later be stained and confused with chromatogram spots. The wide part of the strip wedged itself into the top of the test tube so that no extra support for the paper was required.

A cork stopper was placed in the test tube to prevent evaporation of the solvent. The latter was allowed to migrate up the paper strip for 5 hr., or until it reached to within about an inch of the top of the paper. At the end of this time the paper strip was removed from the test tube, the height of solvent migration indicated by a pencil mark, and the strip dried at about 110°C. for 3 min. The strip was then removed from the oven, sprayed with a solution of ninhydrin reagent and replaced in the oven for four minutes to develop any color reaction between the substances extracted from the glue and the ninhydrin. If colored spots developed that were not less than the size of the original glue spot, then the measured distance of the center of this spot to the center of the glue spot, divided by the distance of ascent of the solvent on the paper above the glue spot, was

designated as the R_f value. Although the indicated breakdown products were not identified, it is known that the R_f value is different for each amino acid and peptide and it was concluded that each spot that was developed represented such a breakdown product of the glue.

9. *Dilution Factor.* There was a possible danger, in the contact angle measurement, of gelation before the drop of glue had come to equilibrium on the strip of paper. If this occurred there would no longer be a liquid-solid interface and the measurement of the contact angle would be erroneous. It was impractical to make contact-angle measurements at 45°C. so the 7.7% dispersions were diluted to produce the same relative viscosity at 25°C. as was measured for the 7.7% size at the higher temperature. This adjustment to the same viscosity was based upon the assumption that spreading and penetration of glue onto and into paper was influenced to some degree by the viscosity.

It was found that different glues required different amounts of dilution to obtain this same viscosity at the lower temperature. This variation was reported as the "dilution factor." Theoretically, the dilution factor should be related to the swelling and gelling properties of a glue and thus have an influence upon sizing efficiency and wet-strength development. Conversely, the more water that is imbibed and held by a glue the more difficult it should be to remove the water from the sized paper in the air-drying process. It has been found from air-drying studies that this must be the case because papers sized with different grades of glue require slightly different drying schedules to obtain a certain moisture content in the paper.

To find how long it would take to test for the dilution factor, the viscosity and density of the concentrated size (7.7% based upon oven-dry glue weight) were first determined at 45°C. Several 20-ml. samples of this glue size were taken and each one diluted with a different amount of water. The amount of dilution was approximated by adding 1 ml. of water per min. of flow time of the viscosity determination. This usually represented the maximum dilution and the other samples of the series were diluted with progressively smaller quantities. The viscosity of each diluted sample was measured at 25°C. until one was found that gave the same flow time as the original concentrated sample at 45°C. The density of this sample was measured so that the relative viscosity might be calculated according to the formula given above. It is to be realized that in making this test three properties of a glue were measured: the relative viscosity, density, and dilution factor.

10. *Imbibition of Water by Glues.* R. H. Bogue (*4*) has stated that one of the most important properties of glue is its ability to imbibe water. The swelling of gelatin and glues under varying conditions, such as pH, has received the attention of many investigators, and their measurements of such swelling have been reported in the literature. The classical method used has been that of measuring the volume change of glues swelled in water at a constant temperature. A simple dilatometric method has been described by Theis and Neville (*5*). This method was used to measure the change in total volume of glue in water during a swelling period of 70 hr. As the glue imbibed water and swelled the total volume of glue and water decreased, a change recorded at certain intervals during the swelling period. The determinations were made in a constant-temperature room at 70°F and 65% R.H. The fine particles of the glue samples were screened out with a 24-mesh sieve and the particles retained used as the test sample. The oven-dry weight of glue sample was adjusted to the size of the swelling chamber or wide-mouth bottle, so that the swelled glue did not fill the bottle, a layer of water remained above the swelled glue, and the end of the capillary tube was not obstructed. Three grams of glue and 86 ml. of water were contained in bottles of about 90-ml. volume. The glue samples were swelled in borax-boric acid-sodium chloride buffer solutions adjusted to pH 7 and also pH 9. When the stopper carrying the capillary tube was inserted in the mouth of the bottle, water was forced up in the capillary tube and the height of the water column measured as the starting point for the determination. Care was taken to avoid entrapment of air bubbles in the glue-water system in the bottle. The capillary tubes of the dilatometer were calibrated and from the calibration curves the change in height of water in the tubes was translated into volume change.

Another attempted control laboratory method consisted of measuring the increase in weight of a known sample of glue exposed to known humidity conditions for increasing lengths of time. This method was also used in measuring not only the amount but the rate of imbibition of water vapor.

Such experiments were made by exposing several glue samples for 48, 72, 144, and 240 hr. to 40, 60, and 80% R.H. These humidity conditions were maintained with the proper glycerol-water solutions in desiccators.

A third possible means of measuring the ability of glue to imbibe water and swell is the determination of changes in viscosity of a glue-water system during the swelling period. Such a method (*6*) gave interesting results in the swelling of guar gums which suggested possibilities for evaluating animal glues. The factors to be considered in the development of such a glue-testing method were: (1) the concentration of the glue-water system; (2) the particle size of the dry glue; (3) the temperature to be maintained; (4) the pH of the system; and (5) a stirring method to prevent formation of a gelled mass in the bottom of the container.

The determinations were run in a constant temperature room at 70°F. The glue-particle size was limited to those passing a 16-mesh and held on a 30-mesh sieve but unless the whole glue sample was ground to this range of particle size, unrepresentative samples were obtained from blended glues. The glue concentration in the system was 5%. The swelling glue was stirred constantly at a rate just sufficient to prevent settling of the particles. If the agitation was too violent, low and erratic viscosity readings were obtained. Stirring was discontinued only for the time required to make a viscosity reading with a Brookfield viscosimeter (Model LVF) using the no. 2 spindle rotated at 30 r.p.m. The particles were allowed to swell in borax-boric acid buffer solutions of either pH 7 or pH 9.

11. *Ash Determination.* An accurate ash determination was made of each glue and this ash analyzed semiquantitatively by the spectroscopic method.

12. *Foaming Test.* In testing the foaming tendency of animal glue by bubbling air at constant pressure through a 50-ml. sample of the size in a 500-ml. gradu-

ated cylinder, it was observed that two zones of foam developed when the time of bubbling air through the glue was extended from a few seconds to several minutes. The first of these zones was a layer of foam composed of small and rather stable bubbles. Pushed up above this first layer were a series of large bubbles that broke easily and constituted no definite layer of foam but did establish a maximum height to which foam could rise in the cylinder. The rate at which air was bubbled through the glue size appeared to have an effect upon the development of foam. The flow rate found to be most effective was 125 ml. per min. Under this condition an equilibrium, in most cases, was established in which the large bubbles in the top layer broke as fast as they were formed. It was also found that the foaming test should be conducted at a temperature high enough to prevent formation of gelled foam through the cooling action of the air flow.

The method consisted of placing a 50-ml. sample of glue size in a 500-ml. graduated cylinder, both the cylinder and the glue sample having been kept at 45 ± 0.5°C. for 2 hr. in the constant temperature bath. Into the cylinder was inserted a long glass tube, to the lower end of which was attached a capillary tube of 0.4 mm. i.d. and bent in the form of a *J* so that the opening pointed up from the center of the cylinder bottom. The tube was placed so that the opening was well below the surface of the 50 ml. of glue size. The top end of the tube was connected with a flow meter which, in turn, was connected to the compressed-air line.

RESULTS OF TEST METHODS

The testing program had two major objectives; the selection of glues satisfactory for tub—sizing of paper and the development of adequate test methods for their evaluation. Obviously, the value of a test method must rest upon its reliability in detecting not only differences in various grades but also possible smaller variations in properties of glues that were rated in the same class. That is to say, there are many glues on the market having a strength rating of about 380 g. and fairly uniform viscosities but, from their behavior as sizing materials, it was suspected that they differed to some extent in other properties. It was one of the purposes of this study to discover those other properties and to measure their significant differences. This required application of the various tests to a large assortment of glues. For this purpose 36 glues from 12 producers were subjected to this preliminary survey. These glues ranged from high gram-strength gelatins to low gram-strength bone glues.

As a result of this preliminary survey nine samples, considered representative of the hide glues, were selected for retesting by the most promising methods and under carefully regulated pH conditions of pH 7 and pH 9. Concentrations of the dispersions were based upon oven-dry rather than air-dry weight of the glue samples.

Softening Point

Although differences were found in the softening or melting points of the glues, there appeared little or no correlation with paper-sizing properties. For this reason there was little justification for continuing the softening point investigations.

Moisture Pickup

Two bone glues and seven hide glues from four different producers were selected for the moisture pick-up determination. Moisture pickup curves were obtained at 80% R.H. for five of these hide glues. If extrapolated to zero-weight increase at zero time of exposure, the curves would show little or no variation in moisture pickup until about 48 hr. exposure time. Thus only the top part of the curves was considered as possibly significant in differentiating glues. The curves for two of the samples fell in the order that might be expected, judging from other known behavior characteristics, and while the differently shaped curves for two other samples made it appear that the quality of glues from one producer might be differentiated, to compare glues from different producers was not valid. Furthermore, it was learned from private communication that different acids were used for acidulation of some of these glues. For this purpose phosphoric acid was used with one glue, hydrochloric acid with two other glues, and sulphurous acid with the remaining two samples. It would be expected that the different calcium salts formed from these acids would produce different deliquescent properties in the glues. Actually, characteristics of bone glues were different from those of three hide glues but similar to those for two other hide glues. But without knowledge of what preservatives or defoamers might have been added or what acids used for acidulation in these samples, it was not considered advisable to use a moisture pickup test to evaluate quality of glues for paper sizing.

Swelling Test

The first dilatometric measurements of swelling made on a series of glues produced results that were in contradiction to other known properties of the respective glues. When some of these glues were retested, fair agreement with the previous tests was obtained in one or two cases but in others the decrease in total volume was a different degree of magnitude from the original test values. The widest variations had been found in blended glues and it was suspected that unrepresentative sampling had been responsible for erratic results. A photographic gelatin, in flake form, was then tested but with no more consistency in the data. Other hoped for improvements in test results were not obtained and it appeared that a simple measurement of volume change was not a reliable quantitative measurement of the swelling of commercial glues.

If the rate at which glue particles swell in water is measured by an increase in viscosity of the glue-water system, a condition obviously limiting the rate of swell-

Table I

Glue Size	*Temp., °C.*	*Foaming, vol. in ml.* — *10 sec.*	*5 min.* — *Dense foam*	*5 min.* — *Total vol. of foam*
Glue—sodium hydroxide	45	400	. . .[a]	500+
Glue—sodium aluminate	45	200	200	325
Glue—meta silicate	45	175	225	375
Glue—"s" sodium silicate (pH 6.94)	45	250	200	400
Glue—"S" sodium silicate (pH 7.92)	45	125	200	300

[a] After 1½ min. of bubbling air through the size the foam went over the top of the 500 ml. graduated cylinder. No readings could be made for more than 5 min. of bubbling time.

Table II

Aging conditions of 7.7% size	*Volume of foam, in ml., at 45°C.*			*Mean*	*Total*
Cooled at 45°C., tested within 20 min.	45/35	45/40	45/40	45/38	83
Aged 2 hr. at 45°C.	45/35	40/40	45/40	42/38	80
Reheated to 60°C., cooled at 45°C. and tested within 20 min.	25/0	30/0	35/0	30/0	30
Reheated to 60°C., aged at 25°C. for 24 hr., then warmed to 45°C. and tested in 20 min.	35/65	45/225	45/165	42/152	194
Cooled to 25°C. for 2 hr. then warmed to 45°C. for 20 min.	40/40	45/45	40/45	42/43	85
Cooled to 25°C. for 2 hr., reheated to 60°C., then cooled to 45°C. in 20 min.	35/25	35/15	35/20	35/20	55

ing is the size of the solid glue particles. In the study of this method for evaluating glue quality, only those particles passing a 16-mesh but retained on a 30-mesh sieve were considered. This selection proved satisfactory for unblended glues but with blended ones, sieving produced unrepresentative samples unless the whole sample was ground to within this size range. In one unground sample, sieving removed the lower-grade glue in the blend resulting in abnormally high viscosity data while in another case the higher-grade glue was apparently separated out and the viscosity readings were unreasonably low.

With the conditions for the test standardized to reduce to a minimum experimental error there were some glues that appeared to swell at abnormal rates and produce viscosity readings that did not correlate with other known properties. It was suspected that defoamers influenced both the rate and magnitude of swelling. Defoamers, being surface-active materials, might be expected to act in some cases as wetting-out agents and accelerate swelling. Glues known to contain various defoamers were tested. In a buffer solution at pH 7 the defoamers decreased swelling during the first four hours of the test but at the end of 6 hr. one of them caused greater swelling than was measured for the glue with no defoamer present. At pH 9 there was a general trend for all of the defoamers present to accelerate swelling of the glue.

The apparent effects of unknown additives in commercial glues upon the phenomenon of swelling, as measured by both the total volume decrease and increase in viscosity, caused both of these methods to be questioned as means of evaluating glues for tub sizing of paper.

Foaming Test

As a test for use requirements, it is essential to know the foaming tendencies of a tub-sizing glue. There are nearly as many foaming tests as there are laboratories concerned with this property. Most of these tests consist of some kind of mechanical agitation with a dip roll or other mechanism to stir a known volume of glue for a limited time, and measurement of the volume of foam created. Such tests are considered unrealistic in that they measure the initial creation of foam but do not measure the total amount of foaming during a sustained period of time. Where size is recirculated from the pump tub to the size press there is a constant mechanical agitation of the glue over a period of hours. Under such conditions the amount of foam that accumulates in the pump tub depends upon two opposing properties: the tendency to foam, and the breaking of the created foam. Unless a complicated miniature recirculation system were built for use in a foaming test it is thought that the foam-creating conditions would be closely approached by an air-bubbling method in which the bubbling would be sustained for a period of several minutes or until an equilibrium between the creation and breaking of foam would be established.

The degree to which a glue foams depends to some extent upon the electrolyte used to regulate its pH condition. In a series of original tests in which three glues were dispersed within the pH range of 8.25 and 8.75 and two others at lower pH conditions with different alkaline materials, the results shown in Table I were obtained.

These results indicated that a 10-sec. foaming test was misleading in that it did not indicate the balance between the creation and the breaking of foams or, in other words, foam stability.

The obviously necessary refinements to this method consisted of bubbling the air through a tube of known small diameter, establishing a known rate of air flow with a flow meter, and keeping the glue-size sample and the graduated cylinder at a known constant temperature.

For the purpose of establishing a standard rate of air flow some experiments were made with two glues, one of which contained a defoaming agent, and the other having no defoamer added. Tests were made with each of these glues at the two conditions of pH 7 and pH 9. Air was bubbled through the glue sizes, at

Table III

Glue	*Aging conditions*		*pH*	*Volume of foam, in ml., at 45°C.*				
	Temp., °C.	*Time, hr.*					*Mean*	*Total*
Sample A	45	2	7.00	25/0	20/0	20/0	22/0	22
	45	2	9.04	15/0	20/0	15/0	17/0	17
Sample B	45	2	7.01	45/0	45/0	45/0	45/0	45
	45	2	9.01	20/0	20/0	20/0	20/0	20
Sample C	45	2	7.00	35/165	45/80	35/75	38/107	145
	45	2	9.00	20/0	20/0	15/0	18/0	18
Sample F	45	2	6.99	20/30	20/25	20/20	20/25	45
	45	2	9.01	10/0	15/0	10/0	12/0	12
Sample G	45	2	7.00	25/105	30/130	30/130	28/122	150
	45	2	9.00	20/0	15/0	15/0	17/0	17
Sample H	45	2	7.01	25/35	25/30	25/30	25/32	57
	45	2	8.99	0/0	0/0	0/0	0/0	0
Sample I	45	2	6.99	0/0	0/0	0/0	0/0	0
	45	2	8.99	0/0	0/0	0/0	0/0	0

7.7% concentration, at two widely different rates, 55 and 125 ml. per min.

It appeared, from these experiments, that the slower rate of air flow was insufficient to create a measurable volume of foam from some glues, particularly at the higher pH condition of 9. And as flow rates higher than 125 ml. per. min. did not give the differentiation obtained with 125 ml. per min., this rate was selected as a standard for the foaming test.

When the reliability of the test was studied by making triplicate determinations of several glue samples, it was found that some uncontrolled condition was causing erratic results. It was then learned that the history of the size, from the time the glue was dispersed at 60°C. until it was tested at 45°C., influenced foam test results. There appeared to be a time-temperature relationship which was explained as hydration equilibrium. If the glue size was allowed to age long enough at the temperature of the test such an equilibrium was established and reproducible results were obtained. This hydration equilibrium could be upset by reheating the size to 60°C. and making the test too soon after the glue had been cooled to 45°C. The equilibrium could be shifted by aging the size for 24 hr. at 25°C. and when retested, excessive foaming was produced. Such changes in equilibrium conditions are illustrated by the results given in Table II, in which the results of each determination are expressed as a fraction: stable foam volume/unstable foam volume.

To be assured that a hydration equilibrium was established, it appeared necessary to hold the glue size at the test temperature of 45°C. for a minimum of 2 hr. This test temperature was selected to prevent incipient gelation of the glue. Prevention of this condition was found to be mandatory in the Jackman-Seawall surface-tension determination, and, possibly, for the same reason. Ross and Butler (7) found that solutions of proteins produce foams of outstanding stability by virtue of "plastic surface films," and that aging the solution or changing the pH with sulphuric acid or with sodium hydroxide, or the addition of small amounts of antifoaming agents, tend to retard or destroy the formation of the "surface-plastic film." This is in agreement with the findings of the authors if it is assumed that surface-plastic film formation is related to the hydration of the glue.

Having standardized the conditions for the foaming test, the foaming properties of a series of glues were determined. These results are given in Table III.

It appeared quite obvious that sample I contained an antifoaming agent and that sample H contained an antifoaming agent that was specific at high pH conditions.

This foaming test was used for evaluating the efficiency of several defoaming agents. The peculiarities of some of the glues tested for foaming tendencies suggested the advisability of determining their surface tensions and ring travel distances. Low surface tension is an accepted prerequisite to foaming but the stability of the foam was found to depend upon the elongation properties of the surface film of glue, as measured by the ring travel distance. For example, a glue that foamed excessively (500 ml.) gave a ring-travel distance of 0.180 in. and a surface tension of 37.2 γ. When 0.1% of a defoamer was added the foam volume was reduced to 23 ml. and the ring travel distance to 0.028 in. The surface tension measurement was 35.4 γ.

In this work it has been found that no accurate appraisal of foaming can be obtained unless the mechanical method of creating foam is sustained for a period of several minutes. Fifteen minutes was found adequate to establish an equilibrium between the rate of foam formation and the breaking of the foam. It was necessary to establish a constant rate of mechanical agitation, which, in the method adopted, was the bubbling of air through the glue dispersion at a constant known rate and through an orifice of known diameter. The pH of the glue dispersion affected the degree of foaming and it was also found that the time and temperature of the interval between glue dispersion and testing was a determining factor in obtaining reliable results.

Table IV

Glue sample	*Gram str.*	*pH*	*Temp., °C.*	*Surface tension* M	*Surface tension* $\gamma = \frac{Mg}{2L}$	*Ring travel after maximum, in.*
A	380	6.98	46.0	0.576	47.10	$43.0 \times 0.004 = 0.172$
		9.03	46.0	0.596	48.75	$43.3 \times 0.004 = 0.173$
B	380	6.98	46.0	0.566	46.25	$42.5 \times 0.004 = 0.170$
		9.00	46.0	0.586	47.80	$47.5 \times 0.004 - 0.190$
C	380	7.02	46.5	0.520	42.48	$41.0 \times 0.004 = 0.164$
		9.03	46.0	0.550	45.00	$44.0 \times 0.004 = 0.176$
D	380	7.02	46.0	0.508	41.50	$43.2 \times 0.004 - 0.173$
		8.99	46.0	0.498	40.70	$14.4 \times 0.004 = 0.057$
E	379	7.02	47.0	0.536	43.80	$18.6 \times 0.004 = 0.074$
		9.03	47.5	0.480	39.20	$8.1 \times 0.004 = 0.032$
F	379	6.99	46.0	0.542	44.11	$10.0 \times 0.004 = 0.040$
		9.00	47.5	0.540	44.11	$9.5 \times 0.004 = 0.038$
G	315	7.00	47.0	0.508	41.50	$13.6 \times 0.004 = 0.054$
		9.02	47.0	0.536	43.80	$10.2 \times 0.004 = 0.041$
H	315	7.06	46.0	0.496	40.50	$44.0 \times 0.004 = 0.176$
		8.99	46.5	0.496	40.50	$20.0 \times 0.004 = 0.080$
I	200	7.00	46.0	0.470	38.38	$7.0 \times 0.004 = 0.028$
		9.00	46.0	0.430	35.12	$7.5 \times 0.004 = 0.030$

M full scale 100 small chart divisions) = 2 g.
g = 980.3.
L = 6.011.
Ring travel, one small chart division = 0.004 in.

Contact Angle and Adhesion Tension

Measurement of contact angles and the calculation of adhesion tensions gave interesting information but such measurements did not appear to predict either sizing efficiency or white spotting of blue prints. If paper was tub sized by the simple process of immersion

then adhesion tension would be of vital importance. The size press, however, not only removes excess glue from the paper surface but also mechanically forces glue into the sheet and accomplishes in a fraction of a second what would take several minutes by immersion. This was proved in making penetration tests of rosin-sized samples of paper (not tub sized) with diluted and stained glue size. With one paper the blueprint solution penetrated in 10 sec. and glue size in 33 sec. With another differently-rosin-sized paper the blueprint solution took 50 sec. to penetrate while the glue size took 192 sec.

Surface Tension. The authors are in agreement with Harkins and Jordan (*2*) that surface-tension measurements by the Du Nouy method left much to be desired. Manual operation of the instrument at a uniform and constant rate was found to be extremely difficult. Furthermore, it was doubted that the surface-tension measurement, in itself, served as a useful criterion of glue-size properties. With the development of the Jackman-Seawell method, hitherto unmeasured filming properties were recorded along with surface tension that proved valuable in interpreting glue-size behavior.

The dome-shaped stress-strain curves obtained by this method using the Instron tester and automatic recorder, are not peculiar to glue testing but have been obtained with starch dispersions, and sugar solutions. With animal-glue sols it was expected that the curves would trail off showing greater ring travel distance with the better grade of glue. However, not all of the supposedly higher-grade glues of about 380-g. strength behaved in this manner. To the contrary, some of them allowed only short ring-travel distance at pH 7 before the ring broke free from the surface (Table IV and Figs 3 to 11). The curves for samples F and G showed nearly identical short ring-travel distance in spite of the fact that sample F had a 379-g. strength rating and sample G a 315-g. strength. It was known that sample F contained two disintegration products that were detected by the chromatographic method and although the quantity of these materials present might not affect gel strength, they could have a decided effect upon the surface- or film-forming properties as indicated by the short ring-travel distance.

Table V

Glue size	*pH*	*Rel. viscosity*	*Diln. factor ml.*	*HCHO req.* *Min.*	*Sec.*	*Ring-travel distance, in.*	*Wet-tensile, g.*	*Variance penetration*
B	6.98	6.92	12.5	8	53	0.178	1302	5.30
	9.03	7.13	12.2	..	..	0.178	1434	8.14
B, 95% Bacto peptone, 5%	6.99	6.54	13.3	12	15	0.172	1257	8.92
	9.02	6.59	11.3	..	..	0.170	1345	10.22
B, 95% Cooked glue, 5%	6.98	6.50	10.55	7	44	0.176 0.162 0.172	1178	7.25
	8.99	6.80	10.50	..	..	0.094 0.088	1254	7.58
B, 90% Cooked glue, 10%	7.02	6.05	11.40	5	56	0.164 0.044 0.056 0.114	1089	6.48
	9.02	6.53	11.00	..	..	0.096 0.062 0.104	1194	8.44

To test the effect of disintegration products in size, two series of experiments were made in which part of the high-grade-glue content of a size was replaced with lower-molecular-weight protein material or peptides. In the first series such a replacement of the B glue was made with Bacto-Peptone and only a very slight reduction in ring-travel distance was found when 5% of the animal glue was replaced by the peptone, although some other properties of the size were adversely affected. In the second series of experiments the substitution was made with a quantity of the B glue that had been cooked at pH 11.5 with limewater for 6 hr. at 96.6°C. It was observed that this lime-cooked glue did not gel when stored under refrigeration at between 0 and 3.33°C. A 5% substitution at pH 7 had no appreciable effect upon ring-travel distance although, like the peptone, other properties of the size were affected. At pH 9 ring-travel distance was definitely decreased. A 10% substitution produced a consistent decrease in ring

Table VI

Glue sample	*Total ash % of sample*	*Ash constituents, % of original glue sample* *Al*	*Ca*	*Mg*	*Zn*	*P*	*Ring travel in in.* *pH 7*	*pH 9*
A	1.2	0.0036	0.36	0.12	NF	F[a]	0.172	0.173
B	3.8	0.0038	1.14	0.38	0.038	NF	0.170	0.190
C	2.9	0.0087	0.58	0.20	0.020	F	0.164	0.176
D	2.8	0.0084	0.84	0.28	0.280	0.084	0.173	0.057
E	3.1	0.0031	0.93	0.03	0.310	0.03	0.074	0.032
F	3.2	0.0096	0.96	0.32	0.32	0.03	0.040	0.038
G	4.4	0.0013	1.32	0.44	0.132	NF	0.054	0.041
H	2.5	0.0250	0.75	0.25	0.250	0.075	0.176	0.080
I	3.7	0.0100	0.74	0.11	1.110	F	0.028	0.030

[a] F = found. NF = not found.

travel at pH 9 and very erratic measurements of this property at pH 7. The results of these two series of experiments are in Table V.

The Jackman-Seawell method gave a different evaluation for some of the glues when they were dispersed at pH 9. Samples D and H had indicated excellent film properties at pH 7 but at pH 9 the ring-travel distance was very short. It did not appear reasonable that these particular glues alone would behave so differently under such a pH change, and it was suspected that some material or additive in them had reacted with the protein to destroy its filming properties. Such a material might be in the nature of a defoamer or a preservative. If the additive was an inorganic compound it should be evident in the ash analysis of the glues. From a comparison of the ash analysis with ring-travel data an answer was found for the peculiar behavior of samples D and H at pH 9. In every case where the zinc content in the glue was over 0.10%, low ring-travel distance at pH 9 was obtained. These comparisons are given in Table VI. Only the major constituents of the ash were selected for this analysis. As was stated previously, the constituents of the ash were determined by a spectroscopic method and are to be considered as semiquantitative.

It is known that zinc is one of several metal ions that react or coordinate with proteins. It has been reported by Neville and Theis (*8*) that zinc acts as a dehydrating agent or prevents swelling of proteins and it has also been listed as a tanning agent and precipitating agent for proteins. The apparent reaction of zinc with protein material might be the cause of the observed increase in cloudiness of some glue sizes prepared at pH 9, as compared with those at pH 7.

Collagen consists largely of glycine, proline and hydroxyproline arranged in helically-coiled protein molecules. The original helical structure is destroyed in the preparation of gelatin and glues. It is not illogical to assume that the resulting irregularly-shaped polypeptide chains tend to revert to a helical configuration. On the surface of a water dispersion of such polypeptide chains there is a boundary layer or "skin" which produces what is termed surface tension. In all probability, this skin is composed of randomly oriented chains but when under stress, as in a surface-tension determination, these chains tend to orient themselves in the di-

Table VII

Glue sample	*Kind of glue*	*Rated gram strength*	*pH*	*Formaldehyde requirement* min.	sec.	*Rf Factor*
J	Hide	600	5.30	3	25	None
			8.74	0		
K	Hide	380	7.48	4	10	None
			8.92	0		
L	Hide	379	7.22	11	45	0.535
			9.31	0		
M	Bone	251	6.75	180	..	None
			8.78	25	..	
N	Hide?	222	7.38	26	30	0.331, 0.457
			8.90	4	25	
O	Bone	222	6.36	37	15	0.224, 0.343, 0.485
			8.62	10	10	
P	Bone	192	6.48	67	5	None
			8.58	15	5	
Q	Hide	192	6.73	10	25	None
			8.88	0		

Table VIII

Glue sample	*Gram strength rating*	*Ash %*	*Rf factor*	*pH*	*Density*	*Rel. viscosity*	*Diln. factor ml.*	*HCHO req.* min.	sec.	*Ring travel, in.*	*Wet tensile, gm.*	*Variance* Pene-tration	*White spots*
A	380	1.2	...	6.98	1.0129	9.15	11.1	2	45	0.172	1218	5.77	0.43
				9.03	1.0135	9.32	10.6	...	..	0.173	1423	19.34	1.22
B	380	3.8	...	6.98	1.0135	6.92	12.5	8	53	0.170	1302	5.30	1.23
				9.03	1.0136	7.13	12.2	..	..	0.190	1434	8.14	0.74
C	380	2.9	...	6.97	1.0124	8.95	12.0	4	12	0.164	1353	4.49	2.04
				9.00	1.0142	8.63	11.6	...	..	0.176	1463	7.39	0.49
D	380	2.8	...	6.98	1.0117	7.35	13.8	3	33	0.173	1435	15.55	0.92
				7.04	1.0148	8.06	12.5	...	..	0.057	1524	12.32	0.97
E	379	3.1	...	7.04	1.0125	8.81	11.9	2	24	0.074	1370	7.18	0.97
				9.02	1.0140	9.28	11.1	..	..	0.032	1430	7.74	1.31
F	379	3.2	0.421	7.02	1.0137	7.55	10.3	4	45	0.040	1257	11.13	0.79
			0.586	9.00	1.0158	7.96	10.2	...	..	0.038	1295	15.00	0.71
G	315	4.4	...	7.06	1.0136	6.75	11.4	6	22	0.054	1281	7.47	0.90
				9.03	1.0143	6.81	11.3	...	..	0.041	1447	22.84	0.90
H	315	2.5	...	7.06	1.0130	6.22	10.0	4	38	0.176	1308	8.22	0.70
				8.99	1.0146	6.15	10.4	...	..	0.080	1457	12.21	0.87
I	200	3.7	...	7.00	1.0136	5.47	9.3	7	0	0.028	1258	6.42	1.16
				8.95	1.0152	5.56	9.0	...	..	0.030	1294	17.87	0.75

rection of the applied stress. Such more or less parallel orientation of the chains increases the surface contact between chains and thus increases the force required to rupture the surface, or, in other words, increases the elastic properties of the surface film.

According to present theory of protein structure, the large coiled and/or helical molecules are held together by side chains that are either acceptors or donors of protons and the number of hydrogen bonded points between molecules is a determining factor in the elastic properties of a protein film. There is a pH range at which bonding is at a maximum for each type of protein. This being true, it appears probable that the reduced elongation, or ring-travel distance, for some glues at pH 9 is a measure of a reduction in hydrogen bonding. It is reasonable to expect such reduction to be caused by high pH conditions, the coordination of polypeptides with metallic ions, and by an acceleration of the effects of defoamers at high pH conditions.

Such theoretical considerations help to explain why the measurement of ring-travel distance, as obtained by the Jackman-Seawell method, has proved to be of value. The data indicated that glues having a ring-travel distance of 0.10 in. or less at both pH 7 and pH 9 produced unsatisfactory sizing.

Formaldehyde Requirement

The formaldehyde requirement test was found to be most useful in detecting bone glues. Any measurable reaction time, i.e., above 1 sec. for the reaction between formaldehyde and glue dispersed at a pH of 9 indicated either very poor hide glue or a bone glue. All of those known to be bone glues possessed long reaction times with formaldehyde. The formaldehyde requirement for one glue, reputed to be of the hide type, was so large at a pH of 9 that it was strongly suspected the sample was a blend of hide and bone glues. The formaldehyde requirements for a few representative glues are given in Table VII.

Dilution Factor

The dilution-factor determination appeared to furnish information beyond that indicated by the viscosity characteristics. Good sizing efficiency was obtained with those glues having dilution factors of 11.5 or greater and poor sizing could be expected if the dilution factor was less than 11. Glues having dilution factors between these two values might or might not produce satisfactory tub sizing. The case of sample A illustrated the value of this determination. Abnormally low ash, high viscosity, low formaldehyde requirement, and long ring-travel distance indicated that this glue might be recommended for tub sizing. However, the low dilution factor at pH 9 served as a warning signal that sizing efficiency at this pH condition might not be satisfactory. When the blueprints were subsequently tested for penetration of coating solution, an abnormally high variance of reflectance readings was obtained, as can be seen from the data presented in Table VIII. Whatever reduced the sizing efficiency of this glue as well as its ability to imbibe water at pH 9 was not detected in either the measurement of ring-travel distance or the chromatographic detection of disintegration products, as in the case of sample F. Both of these glues can be cited as warnings against judging a glue entirely by its rated gram strength and viscosity characteristics.

Viscosity

Viscosity is a very informative characteristic of animal glue, which, at some given arbitrary pH condition gives a rough classification. Further knowledge of glue quality was obtained by comparing viscosity values obtained at the two conditions of pH 7 and pH 9. Unless a higher viscosity was obtained at pH 9 than at pH 7 the glue was held as suspect. For example, the viscosity increase for samples G, H, and I was so small that it was not considered significant and these glues did not develop adequate sizing toward blueprint coating solution, as judged by variance in reflectance readings (see Table VIII). According to viscosity characteristics, sample F should have developed adequate sizing in the paper but the variance at both glue pH conditions was high. Although the formaldehyde requirement of this glue appeared to be satisfactory, the dilution factor and wet tensile were low. The presence of two disintegration products in this sample, as detected by the chromatographic method, offered an explanation for the behavior of this glue which could not be recommended for paper sizing.

In spite of its gram-strength rating, sample E was a borderline glue. Its viscosity, dilution factor, and formaldehyde requirement were satisfactory but its ring travel distance was midway between good and poor. This was reflected in only fair sizing efficiency as shown by variance in penetration. The drop in ring-travel distance at pH 9 indicated high zinc content of the sample.

CONCLUSIONS

It was not surprising to find that in evaluating blended glues no one or two tests served as criteria for the selection of a good glue for tub sizing technical papers. If a glue had been obtained from but one cooking of hide stock, that is, was not blended, and preservatives, antifoaming, or other modifying agents had not been added, its appraisal would be simplified. However, with blended or compounded glues, as received on the market, the data from several kinds of tests, as indicated in Table VII, should be obtained and the results analyzed. To do otherwise deprives the paper mill of information needed for the purchase of satisfactory glues and handicaps the glue manufacturer in meeting the required standards.

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A Study of the Sodium Aluminate-Sodium Abietate Size Precipitates

I. The Composition of the Size Precipitates

ROBERT G. GUIDE

Size precipitates were prepared by acidifying, with perchloric acid, dilute solutions of sodium aluminate and sodium abietate. The compositions of the size precipitates were found to be independent of the pH of precipitation, but dependent upon the initial abietate-to-aluminum mole ratio prior to acidification. The precipitates were not stoichiometric compounds, but were believed to be coprecipitates of several compounds. Free abietic acid was found in all precipitates. Strong evidence which indicates that size precipitates undergo olation during drying was found.

One of the several outstanding properties required of a sheet of paper or board is resistance to water penetration. Theoretically, the penetration of water into a sheet of paper can be prevented or retarded by creating and maintaining fiber surfaces which have sufficiently low free surface energies. In practice, the most common way of doing this has been to precipitate rosin size with papermaker's alum, in the presence of the fibers. This process is called internal rosin sizing.

Various sizing problems confront the practical papermaker. Some mills encounter poor sizing during the summer months or when high temperatures are employed. Others encounter extreme variations in the degree of sizing from day to day or even from shift to shift. The problem of "fugitive" sizing afflicts other mills.

In an attempt to solve these problems, investigations have been conducted to elucidate the mechanism of the rosin-alum internal sizing process. These investigations have dealt chiefly with the physical and chemical properties of the size precipitates and the cellulose fiber. Some of these properties are chemical compositions, electrokinetic potentials, and the contact angles which these substances form with water.

Robinson (*1*), Price (*2, 3*), Jayme and Seidel (*4*) and Back and Steenberg (*5*), have studied the compositions of precipitates formed from the alum-rosin or alum-abietate system. Robinson and Jayme and Seidel believed that an aluminum triabietate existed, but were unable to isolate it. Back and Steenberg, on the other hand, disputed the existence of the triabietate, and concluded that a precipitate which appeared to have the composition of an aluminum triabietate was actually an equimolar mixture of aluminum diabietate and abietic acid. Price (*2*) agreed with this interpretation. In addition, she found no evidence of aluminum hydroxide in any of the size precipitates which were prepared in distilled water.

Robert G. Guide, Graduate Student, The Institute of Paper Chemistry, Appleton, Wis.; present address: Longview Fibre Co., Longview, Wash.
Work performed at The Institute of Paper Chemistry.

Back and Steenberg (*5*), and Ekwall and Bruun (*6, 7*) have studied the contact angles which a water droplet makes with a film of size precipitate. Both pairs of workers agreed that the aluminum salts have lower free surface energies than abietic acid. The salts exhibited contact angles which were 15 to 17° higher than those exhibited by the acid.

Cobb and Lowe (*8*) interpreted the alum-rosin sizing mechanism from the viewpoint of coordination chemistry. They proposed that the aluminum ion is capable of coordinating with any anions in solution. They concluded that such ions as tartrate and citrate are detrimental to sizing because they displace abietate ions from aluminum abietates. They also proposed that the aluminum ion coordinated with cellulosic hydroxyl groups.

Collins, Davis, and Rowland (*9*) and Thode and coworkers (*10*) have studied the nature of the electrokinetic potential of alum-rosin size precipitates. These workers found that both the pH of the suspension and the composition of the precipitate influenced the potentials. Thode and Htoo (*11*) and Ninck Blok (*12*) have studied the electrokinetic potentials of sized and unsized cellulose fibers. They concluded that when cellulose fibers are dispersed in water, they have a highly negative potential, the sign of which is difficult to reverse. Thode and coworkers (*10*) compared the electrophoretic mobilities of size precipitates with their ability to size cellulose fibers. They found that the most rapid increase in sizing efficiency was accompanied by the most rapid increase in the electrophoretic mobility.

In the early 1940's, it was discovered that a portion of the alum used in sizing could be replaced by sodium aluminate. With the use of sodium aluminate, it has become possible for many mills to increase the pH of sizing to as high a level as 7.0, and still maintain satisfactory sizing (*13–17*). Several advantages have been demonstrated in these systems as a result of the increased sizing pH. They are: better and more permanent sheet strength properties, lowered chemical costs, and reductions in paper machine corrosion.

The aim of this investigation was to study some of the chemical and physical properties of sodium aluminate-abietate size precipitates in order to help clarify the mechanism of internal rosin sizing. With the sodium aluminate-sodium abietate-perchloric acid system, it was possible to separate the effects of two variables on certain properties of the size precipitates.

The two variables were: (1) the initial abietate-to-aluminum mole ratio in solution prior to precipitation, and (2) the pH of precipitation.

The properties which were studied were: (1) the

composition of the precipitates, (2) the relative free surface energy of the precipitate as reflected by the contact angle which a droplet of water made on a film of the precipitate, (3) the electrokinetic potential of the precipitate particles, and (4) the ability of the precipitate to size wood cellulose fibers.

The results of this study will be reported in two papers. This paper will discuss the effects of the variables listed above on the composition of the size precipitate. The second paper will discuss the effects of these two variables on the other three precipitate properties listed above. It will also contain a proposed mechanism for the internal rosin sizing process.

EXPERIMENTAL PROCEDURES

Preparation of Size Precipitates

The reactants in this study were sodium abietate, sodium aluminate, and perchloric acid. The reaction medium was deionized distilled water.

Sodium abietate was prepared by saponifying, under nitrogen, pure abietic acid with a 10% excess of 0.3 *N* NaOH. Pure abietic acid was prepared by the method of Palkin and Harris (*18*).

A stable solution of sodium aluminate was prepared by dissolving a reagent-grade mixture of 75% $NaAlO_2$ and 25% NaOH in water. All of the water used in this work was purified to a specific resistance of 1.125 megohm-cm. by passing distilled water through a column which was packed with Amberlite MB-3 mixed resin. The pH of the system was controlled with a 1% aqueous solution of chemically pure perchloric acid.

The size precipitates were formed at an abietate concentration of 0.01% in a 4-l. beaker at room temperatures. The pH of the dilute sodium abietate-sodium aluminate solution was slowly lowered by adding 1% $HClO_4$ from a buret, while mixing the solution with an air-driven stirrer. The addition of the acid was stopped when the desired pH of the suspension was reached. The pH of the system was constantly monitored by means of a Beckman pH meter, using glass and standard calomel electrodes.

The size precipitate was isolated by centrifuging the dilute suspension discussed above. The suspension was passed through a Sharples-type T-1 steam-driven continuous cylindrical supercentrifuge. This centrifuge develops a bowl speed of 50,000 r.p.m., a speed which produces a centripetal acceleration of 61,500 g at the cylinder wall. The rate at which the suspension was passed through the centrifuge varied from 75 to 300 ml. per min., depending upon the particle size of the suspension.

A heavy cellophane liner was placed inside the centrifuge cylinder, and the size precipitate particles were collected on it. The precipitate was removed from the liner and dried in a 40°C. vacuum oven in two steps. These steps consisted of drying over magnesium perchlorate for 18 hr., and then over fresh phosphorous pentoxide for 12 hr. The dry precipitate was then ground up into a fine powder, and stored under anhydrous nitrogen.

Analysis of Size Precipitates

A portion of the dry pulverized size precipitate was extracted with purified acetone to remove any coprecipitated abietic acid. The extract was reserved for analysis, and the residue was dried in a vacuum oven over activated charcoal at 40°C. This residue was weighed and then extracted with a 2:1 mixture of purified chloroform and benzene. The extract, containing aluminum abietates, was concentrated under a stream of nitrogen at room temperature, and the residue from the evaporation was further dried in a vacuum oven at 40°C. over activated charcoal. The dried residue was weighed and stored under nitrogen for further analysis. The residue from the chloroform-benzene extraction was also dried in a vacuum oven over activated charcoal, weighed, and reserved for analysis.

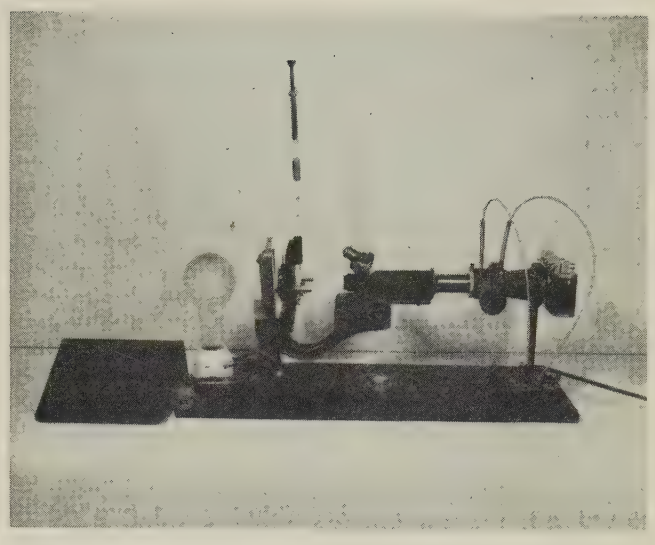

Fig. 1. Apparatus for measurements of contact angles of water droplets on films of size precipitates

All extractions were carried out in 13 by 120-mm. covered test tubes. Approximately 0.5 g. of the size precipitate was extracted with 5 ml. of purified acetone. The same ratio of solids to solvent was used in the chloroform-benzene extraction. After gentle stirring and shaking, the mixture of solids and solvent was centrifuged in a clinical centrifuge at 3500 r.p.m., a speed which produced a centripetal acceleration of 2460 g. The supernatant solution was decanted and the residue was washed with 2 ml. of the pure solvent and recentrifuged. The wash liquid was combined with the main extract. The time of contact between the solids and the solvent was 40 min.

In order to prevent hydrolysis of the aluminum abietates during the solvent extractions, the solvents were carefully purified and dehydrated, and all extractions and manipulations were carried out in a box in which the relative humidity was kept below 4%.

Abietic acid in the isolated size precipitate was determined by a nonaqueous titration according to the general method of Fritz (*19*). Aliquots of the acetone extract were titrated with sodium methoxide, using thymol blue as the indicator.

The abietate contents of both the chloroform-benzene soluble and insoluble fractions were determined by means of carbon analyses. This was possible because the carbon content of the abietate molecule was known, and the abietate molecule was the only source of carbon in these fractions. The analyses were conducted by The Institute of Paper Chemistry microanalytical laboratory according to the method of Niederl and Niederl (*20*).

The aluminum contents of both the chloroform-benzene soluble and insoluble fractions were determined by a photometric technique based on a method described by Sandell (*21*). The samples were dissolved and oxidized by a wet combustion. A buffer solution and the color-forming reagent, ammonium aurintricarboxylate, were then added to the neutralized sample-containing solution. The optical density of the test solution was determined at 520 mμ. The reading was taken exactly 15 min. after the introduction of the ammonium aurintricarboxylate. The instrument used to measure the optical density of these solutions was the Bausch and Lomb Spectronic 20 colorimeter.

Measurements of Contact Angles of Water Droplets on Films of Size Precipitates

Apparatus. The instrument which was used to determine the contact angle between water and a continuous film of the size precipitate is shown in Fig. 1. It was essentially a horizontal microscope through which a photograph of a water droplet resting on a coated glass slide could be taken. The apparatus consisted of the following essential parts (numbers refer to Fig. 1):

1. A 150-w. opaque incandescent lamp.
2. An infrared filter containing a 2% aq. solns. of copper sulphate.
3. A 0.6-cm. diaphragm.
4. A pipet guide.
5. Brackets which held a 1 by 7-cm. glass microscope slide in a level horizontal position.
6. A 48-mm. objective lens.
7. The microscope tube, positioned in the same plane as the glass slide.
8. A Wetzler $^1/_2\times$ adapter which replaces the normal microscope ocular. The front of this adapter fits into the microscope tube and has an eyepiece (9) extending at right angles from its main body. One can view the object through this eyepiece by means of a built-in prism. The back of this adapter is threaded to accommodate a 35-mm. Leica camera (10) whose lens system has been removed. The Wetzler adapter contains both the lens and shutter to be used with the camera body. The camera body is used merely as a film holder.
11. A metal cover for the lamp and filter.

Adox KB 14 black and white film was used throughout this work, and the shutter speed was $^1/_{10}$ sec. There are no adjustable lens openings in the Wetzler adapter. The total magnification obtained with this instrument was 15 diam.

Technique. Approximately 20 mg. of the dried precipitate were dissolved in 2 ml. of purified chloroform. A clean microscope slide which had been split to a width of 1 cm. was flooded with this solution, and the slide was dried *in vacuo* over activated charcoal. This procedure was repeated four times. Finally, the coated slide was placed over activated charcoal in a 40°C. vacuum oven, and kept there for 2 hr.

The photographs of the water droplets were taken in a room where the temperature and relative humidity were kept constant at 72°F. and 50%, respectively. The slide was put in place, and a 3-microliter droplet of purified water was transferred from a micropipet to the coated slide. After 5 sec. of contact between the water and the coated slide, a picture of the droplet was taken. Another picture of the same droplet was taken after 30 sec. of contact. At least two droplets were placed and photographed on each slide.

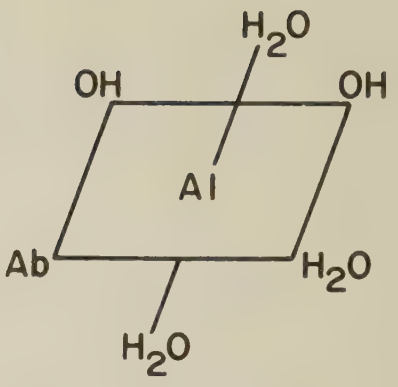

Fig. 2. Probable structure of aluminum monoabietate

The developed films were viewed through a photographic enlarger. Since it is difficult to measure contact angles directly, a method which gives a good approximation was employed. This method, used by Back and Steenburg (*5*), relates the base and altitude of the water droplet to its contact angle with the substrate. If the droplet is assumed to be a part of a sphere, and the effect of gravity is neglected, then,

$$\tan {}^1/_2\,\theta = 2h/b$$

where

θ = contact angle measured within the droplet;
h = height of the droplet above the coated slide; and
b = base diameter of the droplet.

Measurements of Electrophoretic Mobilities of Size Precipitate Particles

The apparatus consisted essentially of a flat microcell through which the particles migrated under an imposed e.m.f., and an ultramicroscope through which this migration was observed. The apparatus and technique were similar to those used by Gorham (*22*). The apparatus differed from Gorham's in the following respects:

1. Platinum wires were used as electrodes.
2. A Cambridge portable potentiometer was used to measure the potential difference in the cell.
3. A simple two-resistor potential divider was placed between the cell and the potentiometer; and
4. The power source was a Reco E-800-2 unit, which contains a full-wave rectifier and filtering elements, and is capable of delivering up to 750 v. d.c., and 200 ma. It is line operated on 110 v. a.c.

Average electrophoretic mobilities were determined for various aluminate-abietate size suspensions. A sta-

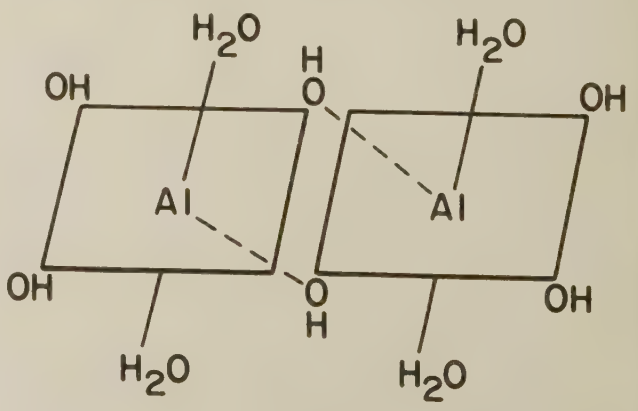

Fig. 3. Probable structure of olated dimer

tistical analysis showed that, within the 95% confidence limits, the error in the average mobilities should not exceed ±7.5%.

Sizing Studies

The pulp used in this work was Weyerhaeuser bleached sulphite. It was extracted with 2:1 benzene-ethanol, then beaten in a 1.5 lb. Valley beater to a S.-R. freeness of 690 ml.

Size precipitates were prepared at the required pH and abietate-to-aluminum mole ratio in the manner already described, but at an abietate concentration of 0.005%. A slurry containing 3.76 g. (O.D.) of pulp was added to the suspension to give a final abietate concentration of 0.004%. The sizing slurry was slowly agitated for 15 min. at a consistency of 0.1%. The concentration of abietate with respect to the fibers was 4.0%.

The treated pulp was diluted to a consistency of 0.004%, and made into 1.88 g. (40 lb. per 3000 sq. ft. B.W.) handsheets on a rapid Kothen sheet mold, without recirculation. Purified water whose pH had been adjusted to that of the sizing slurry was used for dilution.

The sheets were pressed for five minutes at 50 p.s.i., and dried for seven minutes on a steam-heated drying cylinder whose surface temperature was 105°C. The sheets were then conditioned overnight at 72°F. and 50% R.H.

The degree of sizing was determined by the fluorescence size test as recently modified by The Institute of Paper Chemistry (*23*). The porosity of the sheets was determined by TAPPI Standard T 460 m-49, using the Gurley densometer.

EXPERIMENTAL RESULTS

Composition of Size Precipitates

The isolated size precipitates were separated into three components or fractions by selective solvent extractions. Table I gives the results of these extractions.

Table I. Separation of Size Precipitates into Components

Initial[a] *[Ab]/[Al]*	*pH of*[b] *pptn.*	*Acetone soluble, %*	*Acetone-insol. fraction sol. in 2:1 chloroform-benzene, %*
2.0	7.5	21.6	100.0
	7.0	23.7	100.0
	6.5	22.4	100.0
	6.0	22.2	100.0
	5.5	24.2	100.0
1.0	7.5	10.9	100.0
	6.5	11.5	100.0
	5.5	13.5	100.0
0.5	7.5	7.9	42.5
	6.5	9.4	41.4
	5.5	8.6	41.0
0.2	7.5	0.4	0.5
	6.5	0.0	1.6
	5.5	0.5	1.2

[a] This is the mole ratio of abietate-to-aluminum ions existing in the alkaline solution prior to acidification and precipitation.
[b] pH of precipitation.

The abietic acid contents of the isolated size precipitates were determined by sodium methoxide titrations of the acetone extracts. The results are shown in Table II.

Comparison of Table I and Table II shows that virtually all the acetone-extractable fraction of the isolated size precipitate was composed of abietic acid.

The chloroform-benzene soluble and insoluble fractions were both analyzed for aluminum, carbon, and hydrogen. This material had been extracted with acetone prior to being extracted with chloroform-benzene. The results of these analyses are shown in Table III.

Table II. Abietic Acid Contents of Isolated Size Precipitates

Initial [Ab]/[Al]	*pH of pptn.*	*HAb in*[a] *size ppt., %*
4.0	5.5	63.0
3.0	5.5	44.8
2.0	7.5	22.3
	7.0	21.9
	6.5	22.5
	6.0	20.2
	5.5	23.1
1.0	7.5	10.6
	6.5	11.9
	5.5	13.7
0.5	7.5	8.0
	6.5	9.0
	5.5	8.1
0.2	7.5	0.2
	6.5	0.2
	5.5	0.2

[a] HAb refers to abietic acid.

From the data presented in Tables I, II, and III, it was possible to calculate the molar compositions of the isolated size precipitates. These compositions are given in Table IV.

Table III. Analyses of Acetone-Extracted Precipitates

Initial [Ab]/[Al]	*pH of pptn.*	*Chloroform-benzene sol. fraction* Al, %	C, %	H, %	*Chloroform-benzene insol. fraction* Al, %	C, %	H, %
2.0	7.5	5.41	65.83	8.32	...	...	..
	7.0	5.28	67.69	8.51	...	...	..
	6.5	5.47	66.69	8.56	...	...	..
	6.0	5.30	69.79	9.01	...	...	..
	5.5	5.44	65.73	8.53	...	...	..
1.0	7.5	8.47	61.98	8.40	...	...	..
	6.5	8.27	62.40	8.53	...	...	..
	5.5	8.47	60.35	8.32	...	...	..
0.5	7.5	10.10	59.92	8.11	17.20	41.16	6.86
	6.5	10.00	59.68	8.09	17.10	41.34	6.78
	5.5	10.00	59.84	8.13	16.31	40.73	6.72
0.2	7.5	...	...	...	20.50	35.86	6.13
	6.5	...	...	...	20.90	35.13	6.15
	5.5	...	...	...	20.10	35.42	6.10

DISCUSSION

Composition of Size Precipitates

Aluminum acts as an anion exchanger. In a solution of sodium aluminate and sodium abietate, the abietate ion may be expected to exchange with any hydroxyl ion which is combined with the aluminum. These exchange reactions are expressed in equations 1 and 2.

$$[Al(OH)_4]^- + Ab^- \rightleftarrows [Al(OH)_3Ab]^- + OH^- \quad (1)$$

$$[Al(OH)_3Ab]^- + Ab^- \rightleftarrows [Al(OH)_2Ab_2]^- + OH^- \quad (2)$$

As the pH is lowered further, several reactions occur.

$$OH^- + H^+ \rightleftarrows H_2O \quad (3)$$

$$Ab^- + H^+ \rightleftarrows HAb \quad (4)$$

$$[Al(OH)_4]^- + H^+ \rightleftarrows Al(OH)_3 + H_2O \quad (5)$$

$$[Al(OH)_3Ab]^- + H^+ \rightleftarrows Al(OH)_2Ab + H_2O \quad (6)$$

$$[Al(OH)_2Ab_2]^- + H^+ \rightleftarrows Al(OH)Ab_2 + H_2O \quad (7)$$

Assuming that reactions 3 to 7 are governed by similar equilibrium constants, the ratios of the products will be equal to the ratios of the reactants at any pH of precipitation. From this it follows that the compositions

Table IV. Molar Compositions of Total Precipitates

Initial [Ab]/[Al]	*pH of pptn.*	*HAb*	*Al*	*Ab*	*OH*[a]	*(Ab + HAb)/Al*
2.0	7.5	0.51	1.00	1.37	1.63	1.88
	7.0	0.47	1.00	1.44	1.56	1.91
	6.5	0.47	1.00	1.37	1.63	1.84
	6.0	0.43	1.00	1.48	1.52	1.91
	5.5	0.50	1.00	1.35	1.65	1.85
1.0	7.5	0.13	1.00	0.81	2.19	0.94
	6.5	0.15	1.00	0.85	2.15	1.00
	5.5	0.17	1.00	0.80	2.20	0.97
0.5	7.5	0.056	1.00	0.39	2.61	0.45
	6.5	0.057	1.00	0.39	2.61	0.45
	5.5	0.057	1.00	0.40	2.60	0.46
0.2	7.5	0.003	1.00	0.20	2.80	0.20
	6.5	0.003	1.00	0.19	2.81	0.19
	5.5	0.003	1.00	0.20	2.80	0.20

[a] Moles OH obtained by assuming OH = 3 − Ab in the acetone-insoluble fraction. This is justified by the fact that aluminum is a trivalent cation and abietate and hydroxyl are the only anions in this system capable of associating with it.

of the isolated size precipitates will be independent of the pH of precipitation, and dependent upon the initial abietate-to-aluminum mole ratio. It also follows that the size precipitate is not a stoichiometric compound, but a coprecipitate of several compounds.

Because aluminum has a stable coordination number of six, the products expressed in reactions 3 to 7 will contain three molecules of coordinately bound water. The probable structure of the aluminum monoabietate molecule is shown in Fig. 2. This structure is octahedral, in which all the apices are equivalent.

It is known that when alumina sols are dried or aged, they undergo chemical physical alterations which are reflected in such properties as solubility in dilute acids and alkalies, x-ray patterns, etc. According to Weiser (*24*), this gradual change in properties is frequently attributed to a transformation from one allotropic form to another; but, more often, it is due to growth and agglomeration of minute particles into larger granules and agglomerates.

Pfeiffer (*25*) and later Thomas (*26*) proposed a mechanism to account for this agglomeration. These workers hypothesized that molecules of hydrous oxides can be bound by so-called hydroxy bridges. This type of bonding was termed "olation," and the specific hydroxyl bridging as "ol" links or bonds. The olation process proceeds in the following manner.

$$Al(OH)_3(H_2O)_3 + Al(OH)_3(H_2O)_3 \rightleftarrows Al_2(OH)_6(H_2O)_4 + 2H_2O \quad (8)$$

The probable structure of the olated dimer, according to Thomas, is shown in Fig. 3.

The dotted lines in Fig. 3 represent coordinate linkages, and the segment pictured in Fig. 4 is an ol bond. It can be seen that the dimer in equation (8) can continue olating with other monomers or dimers. This process can continue until a close network or agglomerate is formed.

In this study, the concept of olation was extended to size precipitates by assuming that the aluminum abietates are molecules of partially substituted aluminum hydroxide. The aluminum monoabietates may olate in the following manner.

$$Al(OH)_2Ab(H_2O)_3 + Al(OH)_2Ab(H_2O)_3 \rightleftarrows Al_2(OH)_4(Ab)_2(H_2O)_4 + 2H_2O \quad (9)$$

The olated product may have the structure shown in Fig. 5.

The dimer pictured above will have a lower probability than the aluminum hydroxide dimer for continued olation. The monoabietate dimer now has only four possible positions for further ol bridges.

Aluminum diabietates, at best, can only form dimers, or serve as end blocking groups. They may olate in the following manner.

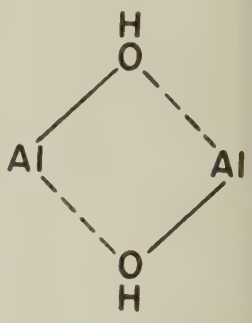

Fig. 4. Ol bond

$$Al(OH)(Ab)_2(H_2O)_3 + Al(OH)(Ab)_2(H_2O)_3 \rightleftarrows Al(OH)_2(Ab)_4(H_2O)_4 + 2H_2O \quad (10)$$

A possible structure for the dimer is pictured in Fig. 6.

Upon further drying, these olated groups of molecules will lose all of their coordinately bound water. This can be explained by the fact that aluminum is also known to have a stable coordination number of four (*27*). The chemical composition of such a completely dried olated

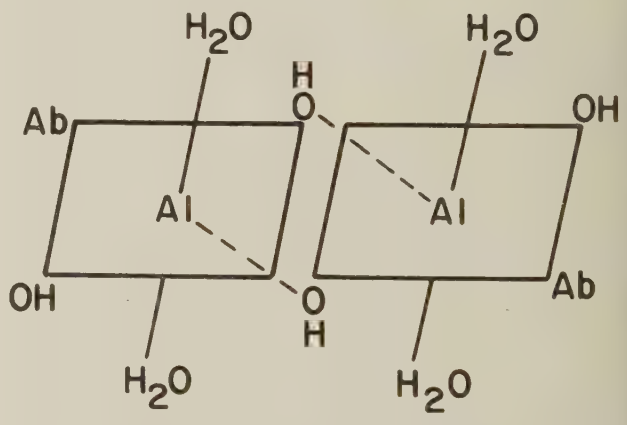

Fig. 5. Probable structure of olated aluminum monoabietate

structure is the same as if it were composed of unassociated anhydrous monomers.

One may visualize how the olation process could proceed in the aging or drying of isolated size precipitates. If the mixture of coprecipitates is rich in aluminum hydroxide, many opportunities would exist for the formation of ol bridges, and many molecules of aluminum abietate would become associated with the aluminum hydroxide. If, on the other hand, the mixture contained very little aluminum hydroxide and mostly abietic acid and aluminum diabietate, very little olation would occur, and the isolated precipitate would consist largely of monomers and dimers.

The occurrence of olation in size precipitates should affect their solubilities in organic liquids. Hildebrand (*28*) has studied the solubilities of nonelectrolytes, and has concluded that the solubility of a polymer is directly proportional to the interaction between it and the sol-

vent, and inversely proportional to the polymer's molecular volume. This relationship can be expressed mathematically in the following manner.

$$S = f(\gamma/V) \tag{11}$$

where

S = some measure of solubility;
γ = the solute-solvent interaction; and
V = the molecular volume of a polymer molecule.

Since aluminum abietates are soluble in chloroform and benzene, the solute-solvent interaction must be appreciable. Aluminum hydroxide, on the other hand,

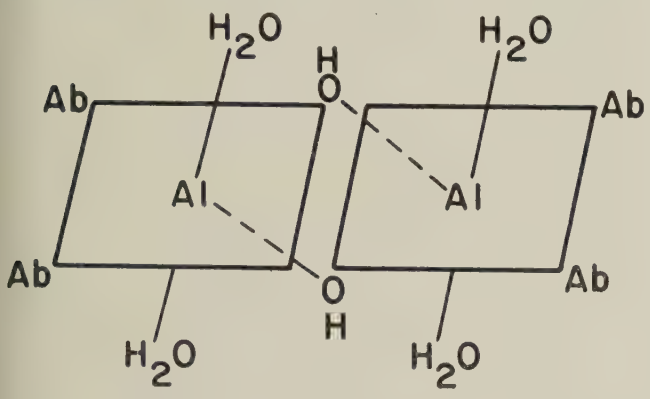

Fig. 6. Probable structure of olated aluminum diabietate

is insoluble in these solvents, and may be assumed to have a negligible interaction with them. A dimer consisting of one molecule of aluminum monoabietate and one molecule of aluminum hydroxide will exhibit approximately the same solute-solvent interaction as the aluminum monoabietate monomer, since the aluminum hydroxide has contributed very little in this respect. The molecular volume of the dimer, on the other hand, will be significantly greater than that of the monoabietate monomer, being equal to the sum of the individual monomer molecular volumes. Equation (11) predicts that such a dimer will be less soluble than the aluminum monoabietate in chloroform or benzene. It follows then, that aluminum hydroxide will tend to reduce the chloroform and benzene solubilities of the coprecipitated aluminum abietates by olating with them.

The following important results were obtained from the size precipitate composition study.

1. The compositions of the size precipitates were independent of the pH of precipitation.
2. No precipitate consisting of a stoichiometric compound was isolated.

Table V. Chloroform-Benzene Solubilities of Acetone-Extracted Size Precipitates

Initial [Ab]/[Al]	[Ab]/[Al] *In acetone-extracted ppt.*	*Theoretically*[a] *soluble, %*	*Actually*[b] *soluble, %*
0.50	0.39	64.1	42.0
0.20	0.20	47.9	1.0

[a] These values were obtained by assuming that all of the abietate must be combined as the aluminum diabietate to yield the lowest solubility. Thus, if the [Ab]/[Al] in the acetone-extracted precipitate is 0.4, 0.4 mole of abietate is present with every mole of aluminum. The abietate is combined with 0.2 mole of aluminum, to give 0.2 mole of aluminum diabetate, leaving 0.8 mole of aluminum to be combined as the hydroxide. By converting the mole ratios of these two compounds to weight ratios, and by assuming that only the aluminum diabietate was soluble, the minimum theoretical solubility was calculated.
[b] Values obtained from Table I.

3. Some of the isolated precipitates exhibited abnormally low chloroform-benzene solubilities.

The initial abietate-to-aluminum mole ratio is an important factor in determining the composition of the size precipitate, whereas the pH of precipitation appears to have little effect. The result was predicted under the condition that the equilibrium constants governing equations (3 to 7) are similar. The absence of precipitates which were stoichiometric compounds was also predicted.

If a size precipitate is composed of unassociated aluminum abietates and aluminum hydroxide, chloroform-benzene should remove the aluminum abietates from the aluminum hydroxide. Thus, theoretically, the chloroform-benzene solubility of any isolated size precipitate is governed by the percentage of aluminum abietates in the coprecipitate. Table V shows the theoretical and actual solubilities of acetone-extracted precipitates.

Table VI. Aluminum Contents of the Chloroform-Benzene Soluble, Acetone-Insoluble Fraction of Isolated Size Precipitates

Initial [Ab]/[Al]	*Al, %*
2.0	5.4
1.0	8.4
0.5	10.0

The wide divergence between the theoretical and experimental solubilities could have been due to either a mechanical entrapment of the aluminum abietates by the aluminum hydroxide or a chemical union between the aluminum abietates and aluminum hydroxide.

Two samples of similar particle size were extracted for 40 minutes and 8 hours, respectively. The 40-min. extraction dissolved 41.3% of the total solids, while the 8-hr. extraction yielded 41.8%. Another sample of the same material was carefully ground into a very fine powder in a small mortar and pestle. This material was divided into two portions which were extracted for 40 min. and 8 hr., respectively. The amount extracted in these cases amounted to 42.5 and 42.9% of the original solids.

Presumably, little or no mechanical entrapment of the aluminum abietates existed. It can therefore be deduced that the only way the normally soluble aluminum abietates were restrained from dissolving was by olating with the insoluble aluminum hydroxide. The effect of olation on solubility was discussed earlier in this paper, where it was postulated that olation between aluminum abietates and aluminum hydroxide would lower the solubility of the aluminum abietates in chloroform and benzene.

From a statistical viewpoint, one would expect this olated structure to consist of polymers in which the mole ratios of aluminum hydroxide to aluminum abietate monomer units would vary over a wide range. Those polymers consisting mostly of aluminum abietates would be soluble in chloroform-benzene. These soluble polymers would contain not only aluminum abietates, but also some aluminum hydroxide.

Of the abietates, the monoabietate contains the most aluminum, 7.45%, whereas the diabietate contains 4.19% aluminum. Table VI shows the aluminum contents of the chloroform-benzene soluble, acetone-insoluble fraction of various size precipitates.

The chloroform-benzene soluble fractions of those precipitates formed at initial abietate-to-aluminum mole ratios of 1.0 and 0.5 all contained more than 7.45% aluminum and must therefore contain some aluminum hydroxide in addition to the aluminum abietates. These results are further evidence of the occurrence of olation in some of the size precipitates.

Although absolute proof is lacking, evidence based on solubility phenomena strongly indicates that the size precipitates undergo olation during drying. As predicted in the theoretical development of this section, the extent of olation was governed by the aluminum content of the size precipitate.

SUMMARY

A study of the sodium abietate-sodium aluminate size precipitates has been described. The following results were obtained when the compositions of the size precipitates were studied.

1. The compositions of the isolated size precipitates formed from solutions of the same initial abietate-to-aluminum mole ratios were independent of the pH of precipitation or isolation.
2. The compositions of the isolated size precipitates were dependent upon the initial abietate-to-aluminum mole ratio.
3. Abietic acid was found in all precipitates.
4. None of the precipitates were found to be stoichiometric compounds, and thus were assumed to be coprecipitates of two or more compounds.
5. No evidence was found for the existence of a triabietate.
6. The isolated size precipitates possessed chloroform-benzene solubilities which indicated the presence of olation within the precipitates.

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A Study of the Sodium Aluminate-Sodium Abietate Size Precipitates

II. The Relationship Between the Physicochemical Properties of the Size Precipitate and Sizing

ROBERT G. GUIDE

Size precipitates were prepared by acidifying, with perchloric acid, dilute solutions of sodium aluminate and sodium abietate. The contact angles of water droplets on films of size precipitates were independent of the pH of precipitation of the size precipitate, but dependent upon the initial abietate-to-aluminum mole ratio in the alkaline solution prior to precipitation. Films consisting mostly of aluminum abietates exhibited contact angles in excess of 90°. Films of free abietic acid exhibited average contact angles of 70.3°. Both the pH of precipitation and the initial abietate-to-aluminum mole ratio influenced the electrophoretic mobilities of the size precipitate particles. Extracted western hemlock sulphite fibers were treated with suspensions of the size precipitates to evaluate their sizing ability. The degree of sizing

Robert G. Guide, Graduate Student, The Institute of Paper Chemistry, Appleton, Wis.; present address, Longview Fibre Co., Longview, Wash. Work performed at The Institute of Paper Chemistry.

increased as the initial abietate-to-aluminum mole ratio decreased, and as the pH of precipitation decreased. Fibers could not be sized at any pH with precipitates having an initial abietate-to-aluminum mole ratio of 0.75 or higher. A theory based on molecular reorientations has been proposed to explain these sizing results. On the basis of the results presented, a mechanism for the sodium aluminate-sodium abietate-perchloric acid sizing process has been proposed.

The previous paper in this series outlined the background and experimental procedures involved in this investigation. It also reported and discussed the experimental results obtained in the study of the composition of size precipitates. This paper will discuss the other properties of size precipitates which were studied, and will propose a mechanism for the sodium aluminate-sodium abietate sizing process.

EXPERIMENTAL RESULTS AND DISCUSSION

Pulp sized with precipitates which had an abietate-to-aluminum ratio greater than 0.75 exhibited no sizing. Even heating the sheets in an oven at 110°C. failed to develop effective sizing.

Contact Angles of Water Droplets on Films of Size Precipitates

The purpose of any sizing material is to prevent or retard the penetration of liquids into a sheet of paper or board. If one analyzes the mechanism of penetration, he must utilize the tools and concepts of surface chemistry. The penetration of liquids into porous media such as sheets of paper or board involves the penetration of liquids through capillaries. Although the actual analysis is very complex because of the irregular shapes and sizes of the capillaries, the principle involved can be explained by the theory of capillarity.

If a liquid surface is curved, the pressure is greater on the concave side than on the convex, by an amount which depends upon the surface tension and curvature of the liquid (*1*). If the curved surface is a part of a sphere, as would be the case if it existed in a uniform circular capillary of radius r,

$$\Delta P = (2\gamma_{LV^\circ} \cos \theta_E)/r \qquad (1)$$

where

γ_{LV° = the specific free surface energy of the liquid, which is mathematically equivalent to the surface tension of the liquid.
θ_E = the equilibrium contact angle between the liquid and the capillary wall.

The rate of penetration of liquids through a capillary may be expressed by the so-called Washburn equation (*2*). This equation combines equation (1) with Poiseuille's equation for laminar flow. It may be expressed as

$$dL/dT = (\gamma_{LV^\circ} r \cos \theta_E)/4\,L\eta \qquad (2)$$

where

L = the length of the capillary filled with liquid or the depth of penetration;
r = the radius of the capillary;
γ_{LV° = the specific free surface energy (surface tension) of the liquid;
η = the viscosity of the liquid;
T = the time of penetration; and
θ_E = the equilibrium contact angle.

Thus, it can be seen that if the liquid and the geometry of the capillaries are kept constant, the rate of penetration becomes a function of the contact angle.

The magnitude of the contact angle is governed by the changes in free surface energy of the solid and the liquid. Young (*3*) first described the free surface energy equilibrium which exists when a drop of liquid is in contact with a smooth solid surface. Later, Bangham and Razouk (*4*) modified Young's equation to read

$$\gamma_{SV^\circ} = \gamma_{SL} + \gamma_{LV^\circ} \cos \theta_E \qquad (3)$$

where

γ_{SV° = specific free surface energy of the solid in equilibrium with the saturated vapor of the liquid;
γ_{LV° = specific free surface energy of the liquid in equilibrium with its saturated vapor; and
γ_{SL} = specific free surface energy of the solid liquid interface.

The contact angle may now be defined as

$$\cos \theta_E = (\gamma_{SV^\circ} - \gamma_{SL})/_{LV^\circ} \qquad (4)$$

From this definition, it is apparent that the magnitude of the contact angle will depend upon the chemical nature of the surfaces concerned. In the case of cellulose and water, γ_{SV° is high, and θ_E approaches zero. Inspection of equation (2) shows that penetration of water into a system of cellulose fibers will be spontaneous and rapid.

To prevent or retard this penetration, the specific free surface energy of the fiber surface must be decreased. One way this can be done is by depositing some sizing material on the cellulose surface. The free surface energy of a unit area of this altered surface can be expressed by equation (5).

$$\gamma(_{SV^\circ})' = A_1\,\gamma_{S_1V^\circ} + A_2\gamma_{S_2V^\circ} \qquad (5)$$

where

A_1 = fraction of the altered surface which is still cellulosic;
A_2 = fraction of the altered surface which is composed of sizing material;
$\gamma_{S_1V^\circ}$ = specific free surface energy of cellulose; and
$\gamma_{S_2V^\circ}$ = specific free surface energy of the sizing material.

The degree to which penetration is retarded is therefore dependent upon the free surface energy of the sizing material, and the fraction of the total area which this material covers. The free surface energy of solids cannot be determined experimentally. However, relative values can be obtained by observing the contact angles which continuous smooth surfaces of the solids

Table I. Contact Angles of Water Droplets on Films of Size Precipitates

Initial[a] *[Ab]/[Al]*	*pH of*[b] *pptn.*	θ_5, *degrees*[c]	δ[d]	θ_{30}, *degrees*
Pure HAb	...	70.3	0.81	64.9
2.0	7.5	90.3	2.16	86.9
	6.5	92.6	0.64	88.7
	5.5	91.4	1.35	87.2
1.0	7.5	93.0	1.17	89.1
	6.5	94.3	1.49	85.6
	5.5	94.5	0.87	92.3
0.5[e]	7.5	92.1	2.46	91.7
	6.5	90.8	4.39	86.6

[a] This is the mole ratio of abietate and aluminum ions existing in solution prior to precipitation.
[b] pH of precipitation.
[c] Subscripts on θ indicate the number of seconds of contact time between the water and the substrate before the measurement was taken. The data reported above are the average of at least four measurements of θ_5 and of at least two measurements of θ_{30}.
[d] Standard deviation among replicate measurements of θ_5.
[e] Because of the low chloroform solubilities of the precipitates with abietate-to-aluminum ratios of 0.5 and 0.2, it was difficult to cast a smooth film from them. This caused a loss of precision and made the significance of the measurements doubtful. Consequently, contact angle measurements on other chloroform-insoluble precipitates were discontinued.

make with a small droplet of a liquid. This has been done with the size precipitates prepared in this study.

All of the size precipitates with the exception of pure abietic acid appear to have very low free surface energies and are capable of producing high and relatively stable contact angles with water (Table I). On this basis, it is predicted that all of these precipitates should be able to size a sheet of paper or board. Before these precipitates can exert their sizing influence upon cellulose fibers, they must be uniformly and densely deposited upon the fibers so that the net free surface energy of the sized fibers is effectively lowered. This aspect of the sizing process will be discussed in the following section.

Electrophoretic Mobilities of Size Precipitate Particles

Many authors have attributed the stability of colloidal dispersions to the electrokinetic potentials of the dispersed particles (*5*, *6*). Briefly, the stabilization mechanism may be visualized as follows: when two particles approach each other closely enough, the van der Waals attractive forces cause them to adhere to each other. If the particles are similarly charged, work must be done in bringing up one particle through the electrostatic repulsion field of the other. The charge on the particles, acting through the electrokinetic potential which is produced in the surrounding fluid, hinders the approach of the particles to the critical distance at which they can adhere to each other. Conversely, if the particles possess opposite electrokinetic potentials, they are attracted to each other.

The electrostatic theory of sizing postulates that the size precipitates are attracted to the fibers by virtue of the relative electrokinetic potentials of the size precipitate particles and the fibers. This theory has been described recently by Thode and co-workers (*7*). In the present study, the electrostatic theory of sizing has been extended by postulating that the relative electrokinetic potentials of the precipitate particles and the fibers may also be an important factor in the mechanism of size retention. The phenomenon of size retention may be treated as a special case of sol coalescence, the coalescence in this case occurring between the size precipitate particles and the cellulose surface. The electrokinetic potentials determine whether the precipitate particles and the fibers can approach each other closely enough for strong but short range attractive forces to exert their influence. These forces may be in the form of van der Waals forces or hydrogen bonds.

The use of sodium aluminate and perchloric acid in this study made it possible to separate the effects of the aluminum and hydronium ions on the electrophoretic mobility of size precipitate particles. The use of these chemicals has also virtually eliminated anionic interference, such as is caused by the sulphate ion if alum is used. Figure 1 shows the effect of pH and initial abietate-to-aluminum mole ratio on the electrophoretic mobility. Figure 2 shows the effect of pH and foreign anions on the electrophoretic mobility of size precipitate particles.

The hydronium and hydroxyl ions most strongly influenced the potential, or mobility, of the size precipitate particles. Also, as the aluminum content of the precipitate increased, the effect of pH on the

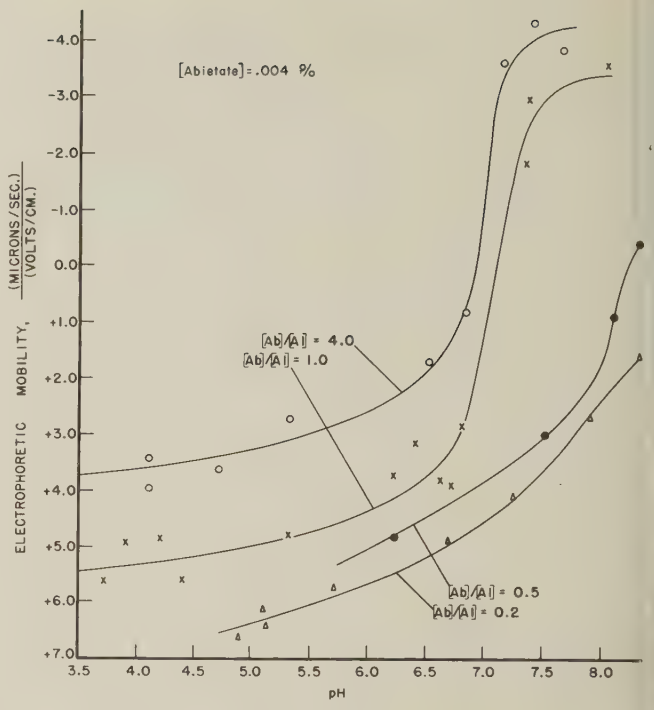

Fig. 1. The effect of pH and initial abietate-to-aluminum mole ratio on the electrophoretic mobility of aluminate-abietate precipitates

electrophoretic mobility decreased. In fact, those precipitates with an initial abietate-to-aluminum mole ratio of 0.2 maintained a positive potential throughout the pH range of 4.9 to 8.2.

Table II shows the relationship between the size precipitate composition and the pH at which the particles will have an electrophoretic mobility of +2.0.

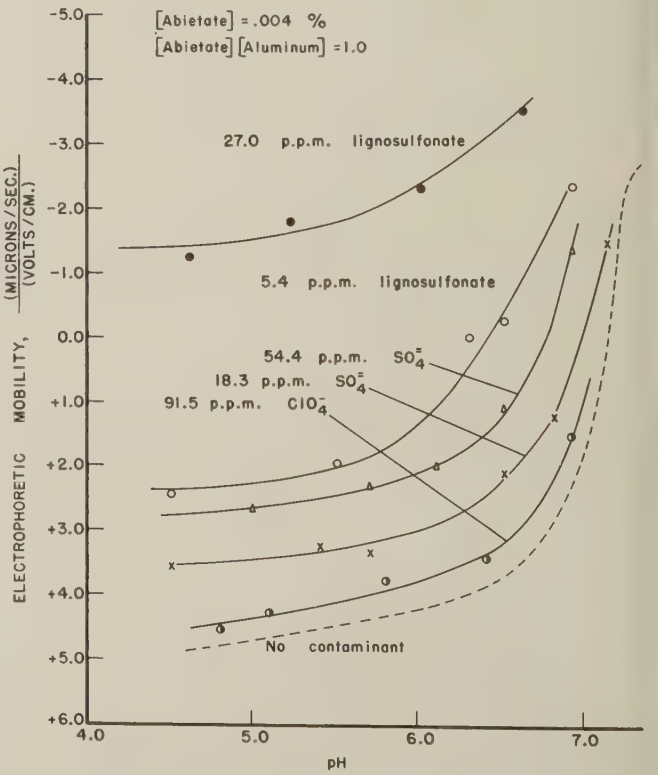

Fig. 2. The effect of anions on the electrophoretic mobility of the aluminate-abietate precipitates

Table II suggests that if one wants to operate at an increased pH, and maintain the same degree of sizing, he must decrease the rosin-to-aluminum ratio in the size precipitate. This ratio, as shown in Table III of the first paper in this series, is nearly the same as the initial rosin-to-aluminum mole ratio.

Table II. The Effect of Composition on the Electrophoretic Mobility of Size Precipitate Particles

[Ab]/[Al] in the ppt.	*pH of pptn. necessary for u = +2.0*
4.0	6.5
1.0	6.9
0.5	7.9
0.2	8.2

Figure 2 is evidence of the effect of foreign anions on the electrophoretic mobility. As was expected because of its very low sorbing and co-ordinating tendencies, the perchlorate ion was virtually ineffective in altering the mobility. On the other hand, only a small amount of a highly electronegative ion such as lignosulphonate was needed to markedly change the mobility. The fact that the sulphate ion lowered the electrophoretic mobilities of size precipitate particles is important because this ion is an unavoidable contaminant when alum is used.

The effect of foreign anions on the electrokinetic potential may have a very important practical significance. The sulphate ion is present in any water which has been treated with alum. In many closed white water systems, the sulphate ion concentration builds up to a very high value. By the same token, the lignosulphonate ion or other lignin residues may be present in appreciable concentrations, especially if the pulp is incompletely washed.

Table III shows the pH at which a size precipitate with an abietate-to-aluminum mole ratio of 1.0 must be precipitated in anion-contaminated process water, if an electrophoretic mobility of +2.0 is to be maintained.

Thus, if sizing is to be successfully carried out at near neutral pH values, anion contamination should be kept at a minimum. The anions undoubtedly fall in a series similar to the Hofmeister series (*8*) in their ability to affect the electrokinetic potential.

The sizing implications discussed above are strengthened by published observations of other workers (*9–13*) who prepared well-sized sheets at pH values between 5 and 7 by incorporating sodium aluminate in their sizing system. In all of these cases, the authors showed that the use of sodium aluminate accomplished two things: (1) a reduction in the sulphate ion concentration, and (2) a reduction in the rosin-to-aluminum ratio. These factors both tend to make the electrokinetic potential of the size precipitate particles more positive and thus presumably tend to increase the size retention on the fibers. Stated in another way, these two factors allow the size precipitate particles to attain a given potential, and a given retention, at a higher pH.

Table III. The Effect of Anions on the Electrophoretic Mobility of Size Precipitate Particles

Contaminant	*Contaminant concentration*		*pH of pptn. necessary for u = +2.0*
	p.p.m.	*Molar*	
None	...	...	6.9
ClO_4^-	91.5	9.3×10^{-4}	6.8
$SO_4^=$	18.3	1.9×10^{-4}	6.6
$SO_4^=$	54.4	5.7×10^{-4}	6.1
Lignosulphonate	5.4	0.2×10^{-4}	5.5

Sizing Studies

Size precipitates which possess both low free surface energies and substantially positive electrokinetic potentials have been prepared. Theoretically, all of these precipitates should be capable of sizing a sheet of cellulose fibers. The ability of these precipitates to prevent water penetration was determined, and the results are shown in Table IV.

From the data of Table IV, it is concluded that the extracted pulp used in these experiments cannot be sized with less than 6% (based on pulp) of size precipitates which have abietate-to-aluminum ratios above 0.75 and which are formed in distilled water. Since it was shown earlier that these precipitates have very low free surface energies, their inability to size the pulp may be caused by two other factors: (1) insufficient or nonuniform deposition of the size precipitate on the fibers, and (2) reorientation of the size precipitate carboxyl groups.

Table IV. Sizing Studies

Initial [Ab]/[Al]	*pH of pptn. and sizing*	*Size[a] time, sec.*
0.75	7.5	0
	7.0	0
	6.5	0
	6.0	0
	5.5	4
0.50	7.5	0
	7.0	0
	6.5	2
	6.0	6
	5.5	22
0.32	7.5	18
	7.0	21
	6.5	35
	6.0	29
	5.5	33
0.20	7.5	13
	7.0	17
	6.5	34
	6.0	38
	5.5	37

[a] Size time expressed in seconds as determined by the fluorescence size test. Two separate batches of pulp were sized at each abietate-to-aluminum ratio and pH of precipitation. Two handsheets were made from each batch of sized pulp, and duplicate size tests were made on each handsheet. The size times reported above therefore are averages of eight separate determinations.

The size retention was determined on some of the handsheets. Two to three grams of the paper were placed in a Soxhlet extractor and extracted with 100 ml. of chloroform for 5 hr. The extract was concentrated, dried in a vacuum oven at 40°C., and weighed. The size retention was calculated by dividing the weight of the extracted solids by the weight of the size precipitate added to the fibers during sizing. Table V gives the results of this study.

These data are qualitative evidence that an increase in the electrokinetic potential of the size precipitate particles is accompanied by an increase in size precipitate retention. This evidence gives strength to the hypothesis that the retention of size precipitate particles is influenced by electrokinetic potentials of the precipitate particles. The data of Table V show that

Table V. Retention Studies

Initial [Ab]/[Al]	pH of pptn. and sizing	Ab during sizing, based on fiber, %	Electrophoretic mobility, u	Size ppt. retained by sheet, %	Size time, sec.
2.0	7.5	3.0	−3.0	10	0
	5.5	3.0	+4.4	27	0
	5.5	6.0	+4.4	47	3
0.5	7.5	4.0	+3.2	9	0
	6.5	4.0	+4.4	13	2
	5.5	4.0	+5.5	35	22
0.2	7.5	4.0	+3.8	18	13
	6.5	4.0	+5.2	33	34
	5.5	4.0	+6.1	37	37

all sheets which were sized at pH 5.5 retained enough size precipitate to impart at least some water resistance to the sheet, if the precipitate was uniformly deposited and anchored onto the cellulose fibers. Nevertheless, those sheets which were treated with precipitates in which the abietate-to-aluminum ratio was 2.0 were virtually unsized.

The fluorescence size testing apparatus is so constructed that the manner in which the water penetrates the sheet can be observed. The penetration was immediate and uniform in all of the sheets which exhibited zero or virtually zero size times. No pinholes and signs of irregular penetration were observed, indicating that the lack of sizing was not due to nonuniform deposition of the size precipitate. Since insufficient or irregular deposition of the size precipitate did not appear to be responsible for the lack of sizing in some of these sheets, it was deduced that the size precipitate carboxyl groups underwent reorientation upon contact with water.

Molecular orientations at solid surfaces greatly influence contact angles. Many molecules contain both hydrophobic and hydrophilic groups. Excellent examples of this are the fatty or resin acids. If the surface contains many carboxyl groups, the contact angle with water will be low. If, on the other hand, the carboxyl groups are buried in the solid, and remain there, the water will exhibit a stable high contact angle.

When either a mono- or a multimolecular film of fatty or resin acids is cast onto a solid surface, the majority of the carboxyl groups will be oriented inward, since that insures the lowest free surface energy. When water comes in contact with such an oriented surface, the natural tendency is for the carboxyl groups to reorient themselves so that they will be as close to the water as possible. This is a result of the strong polar or van der Waals forces existing between the carboxyl groups and water. Langmuir (*14*) called attention to this phenomenon when he showed that the contact angle of a monolayer of a fatty acid with a drop of water varied from 18 to 86°, depending upon the substrate on which the monolayer was deposited. Rideal (*15*) showed that such differences in the contact angles resulted from the differences in the attraction or strength of anchorage between the carboxyl end of the fatty acid and the solid substrate. Studies at The Institute of Paper Chemistry (*16*) have indicated that anchorage of size precipitates on cellulose fibers may be an important factor in the sizing of paper.

When a size precipitate dries on a fiber surface, the precipitate molecules probably orient their carboxyl groups away from the air-size precipitate interface so that the interface will have the lowest free surface energy. The molecules located near this interface are blocked from the fibers and cannot be anchored to them. Yet, reorientation of their carboxyl groups must be prevented if good sizing is to be maintained. It is proposed that the size precipitate molecules are immobilized by a mechanism of olation.

The mechanism of olation was discussed in the first paper of this series, and it was postulated that isolated size precipitates olate upon drying. If olation occurs during drying of the isolated size precipitates, then it must also occur during drying of the size precipitates on cellulose fibers.

The net effect of olation is to form strongly associated macromolecules. These macromolecules will be much more immobile by virtue of their size than the single molecules of aluminum abietates. This immobility of the size precipitate molecules will then retard reorientation of the hydrophilic groups toward the water-sized fiber interface, and good sizing will result.

SUMMARY

The following results were obtained from a study of the sodium aluminate-abietate size precipitates:

1. Droplets of water on smooth films of precipitates with an over-all abietate-to-aluminum mole ratio of 1.0 formed average contact angles of 93.9°.

2. Droplets of water on smooth films of precipitates with an over-all abietate-to-aluminum mole ratio of 2.0 formed average contact angles of 91.4°.

3. Droplets of water on smooth films of pure abietic acid formed average contact angles of 70.3°.

4. Smooth films of precipitates with over-all abietate-to-aluminum mole ratios of 0.5 and less could not be cast, resulting in an inability to obtain precise contact angle measurements on these samples.

5. As the pH of the size precipitate suspension was increased, the electrophoretic mobility of the particles became more negative, for precipitates of constant abietate-to-aluminum mole ratios.

6. As the abietate-to-aluminum mole ratios of the precipitates were increased, the electrophoretic mobilities of the particles became more negative, at constant suspension pH values.

7. The effect of all the anions studied was to make the mobilities of the size precipitate particles more negative. The magnitude of the effect was dependent upon the sorption tendencies and concentrations of the contaminant anions. A suspension of a precipitate with an abietate-to-aluminum mole ratio of 1.0 which was contaminated with 27 p.p.m. of sodium lignosulphonate failed to exhibit a positive electrophoretic mobility at any pH.

8. For size precipitates with the same abietate-to-aluminum mole ratio, the degree of sizing tended to increase as the pH of sizing decreased.

9. Fibers could not be satisfactorily sized at any pH with precipitates having an abietate-to-aluminum mole ratio of 0.75 or higher.

10. The best sized sheets were those which were treated with precipitates having an abietate-to-aluminum mole ratio of 0.2. This was the lowest ratio employed.

11. The amount of precipitate retained by the fibers

increased as the pH of sizing decreased and as the abietate-to-aluminum mole ratio of the precipitate decreased.

CONCLUSIONS

The following conclusions are based on results obtained in a study of the sodium abietate-sodium aluminate-perchloric acid sizing system. These conclusions may also be applicable to the sodium abietate-alum sizing system.

1. Contrary to the conclusions of some authors who have studied the sodium abietate-alum sizing system, isolated size precipitates are not stoichiometric compounds.
2. Compositions of isolated size precipitate are independent of the pH of precipitation or isolation.
3. Compositions of isolated size precipitates are dependent upon the abietate-to-aluminum mole ratio initially existing in the alkaline solution.
4. Contrary to the conclusions of some authors who studied the sodium abietate-alum sizing system, aluminum triabietate is not a part of the size precipitate.

The following conclusions verified results obtained by other workers who studied the sodium abietate-alum system:

1. The electrophoretic mobility of size precipitate particles is dependent upon at least three factors: (a) composition of the precipitate, (b) pH of the suspension, and (c) the presence of other ions in the suspension.
2. Films of aluminum abietates exhibit a much lower free surface energy than do films of abietic acid, based on the contact angles of water on these films.

The following assumptions have been strengthened by evidence obtained in this study:

1. The retention of size precipitates on wood cellulose fibers is a strong function of the electrokinetic potential of the precipitate particles.
2. The size precipitates are coprecipitates of abietic acid, aluminum di- and monoabietate and aluminum hydroxide.
3. In order to size wood cellulose fibers, the size precipitate must contain aluminum abietates and some aluminum hydroxide. Free abietic acid is not beneficial.
4. Coprecipitated aluminum hydroxide and aluminum abietates olate upon drying to yield a network of associated molecules.

A PROPOSED MECHANISM OF THE ALUMINATE-ABIETATE SIZING PROCESS

Any rosin sizing mechanism must consider at least three steps: (1) formation of potentially low free surface energy precipitates, (2) deposition of these precipitates onto the cellulose fibers, and (3) conversion of the wet size precipitate-fiber surface to a stable low free surface.

Based on the experimental results obtained in this study, a mechanism for the sodium abietate-sodium aluminate-perchloric acid sizing process is hereby proposed. The general principles should be applicable also to the commercial sodium rosinate-alum system.

Formation of Precipitates (Step 1)

An alkaline solution of sodium abietate and the aluminum ion contains several ionic species. These may include such ions as abietate, aluminate, and basic aluminum abietates. Upon the addition of acid, these ions begin to precipitate simultaneously as abietic acid, aluminum hydroxide, and aluminum abietates. Further addition of acid increases the amount of material precipitated, but does not change its composition. The composition of the coprecipitate is determined by the abietate-to-aluminum mole ratio existing in the solution at the instant of precipitation, and is independent of the pH of precipitation.

Coprecipitates rich in aluminum abietates are potentially good sizing agents, because these compounds exhibit a low free surface energy when dried. Abietic acid has a higher free surface energy, and thus is not as good a sizing agent as the aluminum abietates. A favorable composition can be maintained by controlling the initial abietate-to-aluminum mole ratio.

Deposition of Precipitates (Step 2)

The electrokinetic potentials of the size precipitate particles and cellulose fibers are the most important forces governing the deposition of the precipitates onto the fiber. When these two solid surfaces possess electrokinetic potentials which are opposite in sign, they are attracted to each other, and the size precipitate is deposited on the fibers. If, on the other hand, the sign of the potentials is the same, and the absolute values of the potentials are high enough to overcome thermal and mechanical agitation, the precipitate particles are repelled from the fiber surfaces, and little deposition occurs.

The pH of the sizing suspension and the presence of foreign ions greatly influence the electrokinetic potentials. Cellulose fibers exhibit a highly negative potential, the sign of which is difficult to reverse. Ionic contamination and pH greatly affect the potential of size precipitate particles and can reverse the sign of the potential. By controlling the ionic environment of the sizing suspension, size precipitate particles with high positive potentials can be produced. Factors which assure a high positive potential are (a) a low initial abietate-to-aluminum mole ratio (0.5 or less), (b) a low pH of sizing (6.0 or less), and (c) a low concentration of easily sorbed anions in the sizing suspension.

Conversion into Low Free Energy Surfaces (Step 3)

Once the size precipitate is deposited on the fibers, the wet surface must be converted into one which will have a stable low free energy, giving rise to a system of effectively sized fibers. This conversion process occurs during the drying of the paper. Molecules of aluminum hydroxide, aluminum abietates, and cellulose hydroxyl groups associate by a mechanism called "olation." The olation immobilizes the individual precipitate molecules so that they are effectively bound to the cellulose fibers, and prevents the hydrophilic groups of the surface molecules from reorienting themselves to create a surface with a higher free energy.

The presence of aluminum hydroxide and aluminum monoabietate in the size precipitate is necessary for effective olation. This is accomplished by maintaining a low initial abietate-to-aluminum mole ratio. The absolute value of this ratio depends upon the system

under consideration; in the case of the sodium abietate-sodium aluminate-perchloric acid system, this value is less than 0.75.

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A Study of the Water Retention of Latex-Bound Pigmented Coating Colors

R. L. HAGERMAN, R. G. JAHN, and W. H. SOMERS

An investigation of the factors affecting the water retention of coating colors containing synthetic latex as the principal binder was carried out. The investigation, which was designed to outline the relative importance of several coating color variables, studied the effects of adhesive composition, adhesive level, per cent solids, type and quantity of synthetic thickener, pigmentation, and order of mixing on water retention and other coating color properties. The most important effect observed in the compositions studied was the increase in water retention due to the inclusion of small quantities of synthetic thickeners. Quantitative relationships between most of the factors studied and the properties measured were expressed in the form of equations derived by statistical methods.

There is a current trend in the paper industry toward the use of higher proportions of latex in paper coatings and, especially, in paperboard coatings. Without going into detail, the object of using higher proportions of latex in pigmented paper and paperboard coatings is to produce a higher quality printing surface. With the increased usage of latex in paper coatings, it was found that as the proportion of latex in the paper coating increased, the water holding power of the coating decreased. This change is easily understood. The synthetic latex particle is hydrophobic and rather than "holding" the water in the coating color, allows it to drain away into the paper substrate. Protein and starch adhesives, on the other hand, are hydrophilic molecules and show a pronounced tendency to "hold" the water in the coating color upon application to paper.

The importance of water retention has been discussed by Frost (*1*) and Culp (*2*). These authors point out that coating colors with low water retention values may show pronounced patterning when applied to paper on roll type coating equipment. The patterning is caused by rapid loss of water into the paper web resulting in undesirable rheological properties. Garrett, *et al.* (*3*), have quantitatively described the leveling of latex paint films on a porous substrate as a function of the water holding and rheological properties of the paint.

Water retention measurements have also been of secondary importance as a tool in understanding some of the effects of coating color variables on some of the properties of coated paper. For example, those properties of the coated paper which are related to the surface smoothness may be correlated with water retention because of its effect on the degree of roll pattern present. Other properties of coated paper, such as pick resistance, are affected by the distribution of the adhesive in the dried coating. Frost (*1*) and Stinchfield (*4*) have shown a relationship between water retention and other properties of paper coatings using starch or casein as the adhesive. In addition, there are in the literature several briefer references to the relationship between water retention and adhesive redistribution in paper coatings (*5–11*).

It seems essential to study the effect of coating color variables on the water retention of latex bound paper coating colors and find means of increasing the water retention of these colors. It should also be useful to determine whether a relationship exists between the water retention of latex bound coating colors and the properties of paper or paperboard coated with these colors. Such a relationship should provide information concerning the redistribution of the latex adhesive in the paper coating.

EXPERIMENTAL

The pigment dispersions used in preparation of coating colors for this investigation were prepared as follows: pigment was dispersed in a Hobart mixer using 0.3% tetrasodium pyrophosphate based on the dry weight of pigment, and a small quantity of water. The resultant dilatant paste was kneaded for 5 to 10 min. and diluted

R. L. Hagerman, R. G. Jahn, and W. H. Somers, Coatings Technical Service, The Dow Chemical Co., Midland, Mich.

PER CENT Solids	PER CENT Adhesive	PER CENT LATEX 80	85	90	95
41	14	X		X	
	16	X		X	
	18		X		X
	20		X		X
44	14		X		X
	16		(X)		(X)
	18	(X)		(X)	
	20	X		X	
47	14	X		X	
	16	(X)		(X)	
	18		(X)		(X)
	20		X		X
50	14		X		X
	16		X		X
	18	X		X	
	20	X		X	

X Combinations Tested
(X) Combinations Duplicated

Fig. 1. Design for first phase

with water to 70% solids. Aliquot portions of this pigment dispersion were mixed with the required amount of adhesive, synthetic thickener, and dilution water. After mixing, the coating colors were filtered through a 115-mesh screen. Before testing, the completed coating colors were allowed to reach equilibrium under standard conditions of 75°F. and 50% R.H. When necessary, the coating colors were de-aired on a laboratory centrifuge before testing. The synthetic latex used as adhesive in this work was a styrene-butadiene latex which is in common usage in the paper coating industry. Viscosities were determined with a

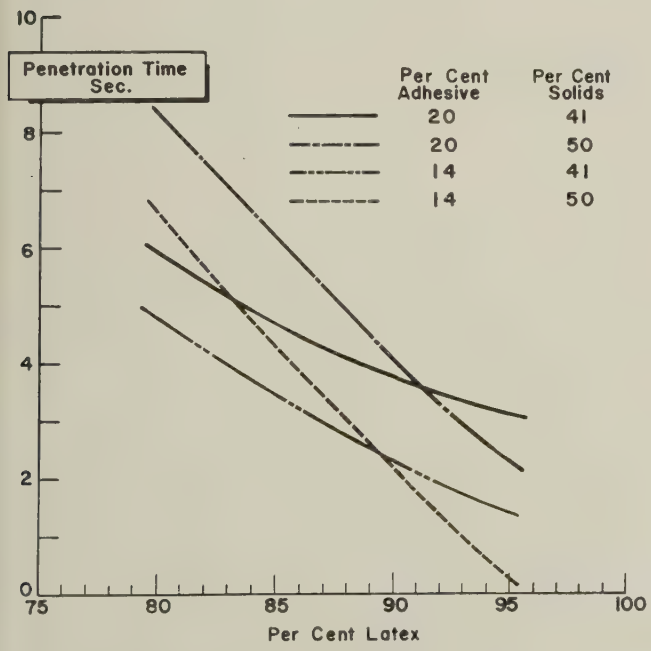

Fig. 2. Effect of adhesive composition on water retention

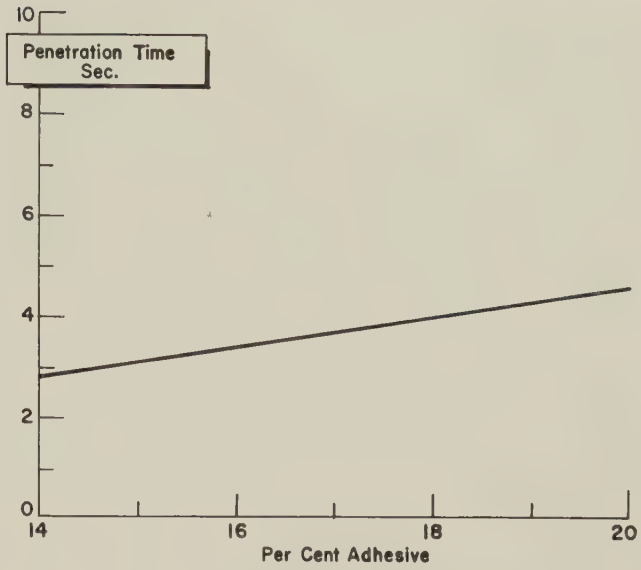

Fig. 3. Effect of adhesive level on water retention; average solids and per cent latex

Brookfield LV viscometer at 75°F. Measurements were made at 60 r.p.m. wherever possible, using a spindle suitable to the viscosity of the color being tested.

Water retention was tested on "standard water retention test paper" supplied by the S. D. Warren Co. using a test apparatus essentially as described by Stinchfield, *et al.* (*4*). The technique was basically as follows: a weight, connected to a milliammeter through a battery, was placed in a puddle of coating color on top of a sheet of test paper which in turn was resting on a flat metal plate. The plate was connected to the other side of the milliammeter. As the water penetrated the sheet, the milliammeter indicated the passage of current. The time interval from the start of this test until the current flow reached a value of 0.5 ma. was recorded. The procedure was modified slightly by placing the coating color on the bottom of the weight rather than on the paper at the beginning of the test. The time was recorded starting when the weight was placed on the paper. Because of the rather short penetration times encountered with the coating colors tested, this modification was thought necessary to yield more reproducible results.

The coating colors were applied to 51-lb. bleached sulphite stock. Coating weight was 15 to 17 lb./TAPPI ream. Prior to testing, the coated papers were supercalendered on a laboratory supercalender and conditioned for 24 hr. under TAPPI Standard T-402 m-49 conditions.

Pick tests were run on an IGT printability tester using an IPI No. 7 tack-graded ink. The end point was determined microscopically (26 magnification) as the first non-isolated pick.

The data obtained were fitted to the equation,

$$y = a + bx_i + cx_j + \ldots + mx_i^2 + nx_j^2 + \ldots + px_ix_j + \ldots (1)$$

by means of a multivariate regression analysis. The x's represent the values of the coating color variables investigated and y is the predicted value of the property measured. The coefficients of equation (1) were calculated on an IBM Model 650 computer. An essential feature of the calculations performed on the

computer was that each of the terms in equation (1) was tested to determine how important it was in describing the total variation of y over the range of variables studied. The unimportant, or nonsignificant, terms were eliminated from the calculation of the coefficients in equation (1). The advantages of treating the data in this fashion are several. The most important one is that each point on the curve or curves drawn from the equation is as reliable as if it were an average of almost as many measurements as were made for the entire experiment.

RESULTS AND DISCUSSION

The major portion of this study consisted of three factorial experiments and it is most convenient to discuss these tests and the results in terms of the individual experiments or phases.

Phase I

The first experiment consisted of the factorial design shown in Fig. 1. This experiment was run as a half replicate using the combinations of factors denoted by an x in the figure. Those combinations of factors which are circled were run in duplicate to provide an estimate of the error in the experiment. The duplicates consisted of separately prepared samples of coating colors for those indicated. The differences between these duplicates would give an estimate of the errors involved in preparation of the coating colors, as well as random errors associated with the measurements of the properties studied.

The following equations for the responses studied were obtained.

$$y_1 = 795.6 - 19.29\,x1 + 0.1102\,x_1^2 + 0.0309\,x_3^2 \quad (2)$$

$$y_2 = 12.89 + 0.0037\,x_1^2 + 0.022\,x_3^2 + 0.0034\,x_1x_2 - 0.022\,x_1x_3 \quad (3)$$

$$y_3 = 3618. - 130.1\,x_1 + 0.7575\,x_1^2 + 107.1\,x_2 + 38.71\,x_3 - 1.849\,x_2x_3 \quad (4)$$

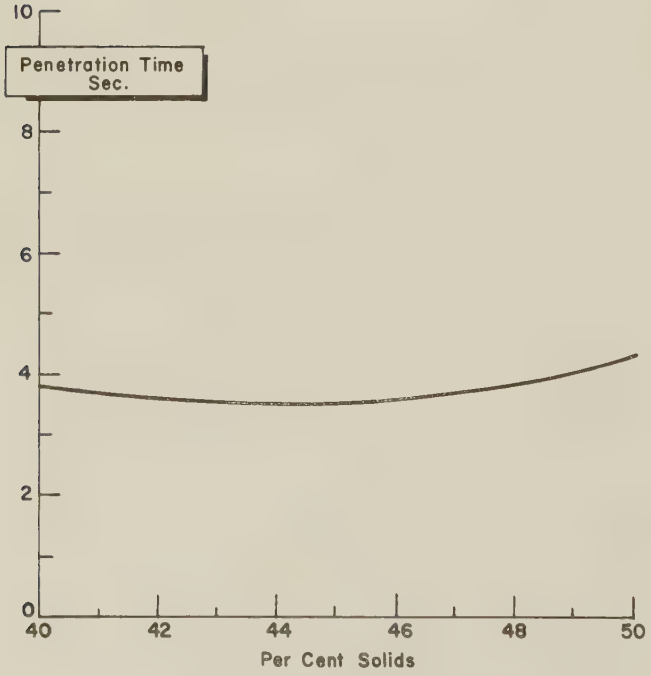

Fig. 4. Effect of per cent solids on water retention; average adhesive level and per cent latex

where

y_1 = Brookfield viscosity, centipoises at 60 r.p.m.
y_2 = penetration time, sec.
y_3 = IGT picking velocity, f.p.m.
x_1 = per cent latex in adhesive (remainder is casein)
x_2 = adhesive level (parts per 100 parts clay)
x_3 = per cent solids

Figure 2 shows equation (2) in graphic form as water retention plotted against the adhesion composition ex-

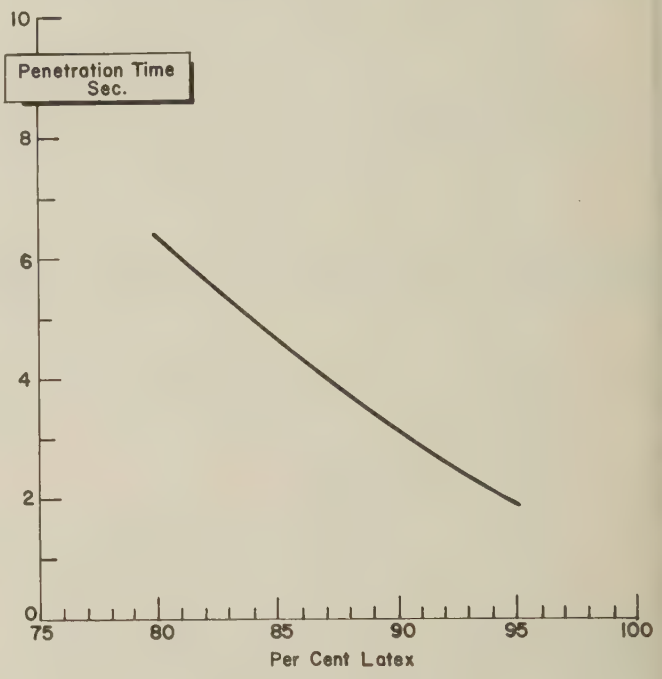

Fig. 5. Effect of adhesive composition on water retention; average adhesive level and per cent solids

pressed as per cent latex. Curves are shown only for the extremes of adhesive level and per cent solids. It will be noted that according to these curves (and the equation), at the higher levels of latex in the adhesive, and increase in the per cent solids causes a reduction rather than the somewhat expected increase in water retention. It did not seem reasonable to attempt an interpretation of this without more detailed experimentation. Since this study was intended merely to outline those properties affecting water retention, no explanation was attempted. To present a simple picture of the water retention test results in this experiment, the effects of the three independent variables are shown in Figs. 3, 4, and 5. In each figure, the water retention (penetration time), is plotted against one of the independent variables with the values for the other two independent variables being held constant at their averages. It is seen that per cent solids has a slight effect under these conditions, the adhesive level has a slightly greater effect on the water retention, and the largest effect of all is found as the proportions of latex and casein in the adhesive are varied.

As equation (2) shows, the adhesive level had no significant effect on viscosity. This would be expected at the levels of latex present in the adhesive used in this experiment, as judged by the work of Erratt, *et al.* (*12*).

Figures 6 and 7 show the effect of per cent solids on pick resistance at, respectively, the 80 and 95% level, of latex in the adhesive.

In Fig. 8, the water retention of each coating color is

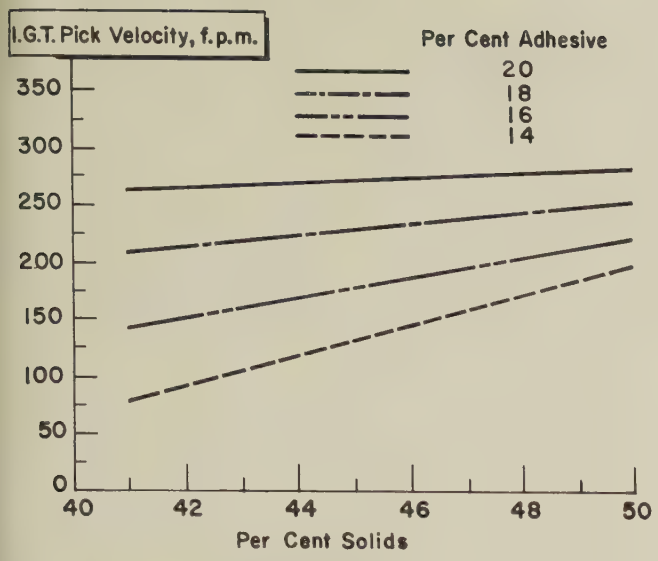

Fig. 6. Effect of per cent solids on pick resistance; 95% latex in adhesive

plotted against the corresponding pick resistance value. In order to make any correlation present more readily apparent when examining the graph, the values plotted were those determined from equations (2) and (3) divided by the standard deviation of each response. When plotted in this fashion, if a significant linear correlation existed, the slope of the best straight line through the points would be a 45° line. It is evident from Fig. 8 that the data show no such correlation, in general. If the points of Fig. 8 are grouped on the basis of the proportion of casein in the adhesive and the correlation of each of these groups measured individually, the following correlation coefficients are obtained:

% Casein in adhesive	*Correlation coefficient*
5	0.10
10	0.17
15	0.58
20	0.77

A correlation coefficient of 1.0 indicates perfect correlation between the factors being studied.

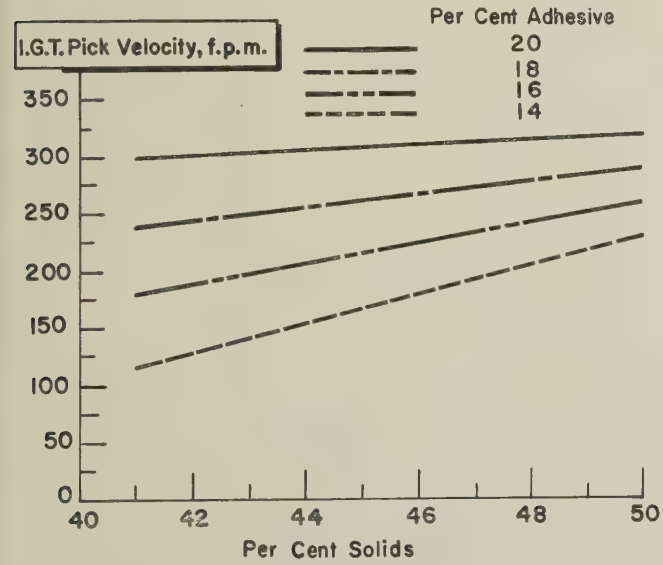

Fig. 7. Effect of per cent solids on pick resistance; 80% latex in adhesive

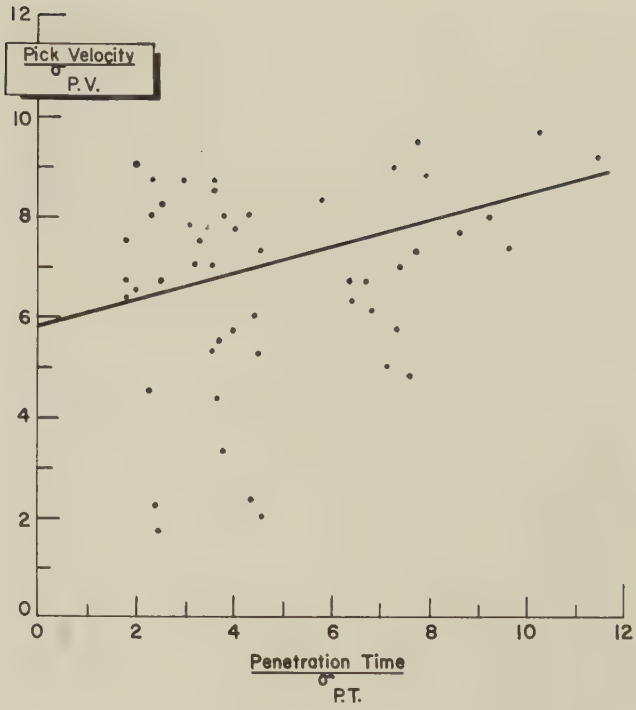

Fig. 8. Correlation of pick resistance with water retention

This figure shows that as the hydrophobic nature of the adhesive is increased by replacement of casein with latex, the pick resistance shows much less correlation with water retention.

Phase II

To evaluate the effect of various modifiers on the water retention of a latex-bound coating color, the materials shown in Table I were used to modify the following coating color:

	Parts by weight, dry
Medium fraction coating grade clay	100
Latex	15
Modifier	0.3
Water to 55% total solids	

The penetration times obtained with these colors are shown in Table I together with the corresponding viscosities. It is of interest to note that the three materials which were effective in increasing penetration time (methylcellulose, carboxymethylcellulose, and sodium alginate), were all carbohydrate derivatives. Starch and starch derivatives were not evaluated because the wide use of starch-latex combinations in paper coating and the wide variety of starches available warranted a separate, complete investigation. The lack of correlation between viscosity and penetration time is evident from a comparison of these properties as shown in Table I.

The effects of the effective synthetic thickeners were investigated in somewhat more detail in the second and third phase of this study. The second phase consisted of a two-thirds replicate of a factorial experiment. The variables included were: type of modifier (methylcellulose versus sodium alginate), pigmentation (clay/calcium carbonate versus clay/titanium dioxide), pigmentation (100, 90, 80% clay in pigment), and per

cent solids. The responses and method of analyzing the data were the same as for the first phase. In this second phase, however, none of the independent variables had a significant effect on the water retention. Perhaps the most important point in this result is that there was no difference in water retention between colors modified with methylcellulose and those with sodium alginate. Because of the lack of effects, this experiment will not be discussed in further detail.

Table I. Effect of Thickeners on Viscosity and Water Retention

Thickener	*Brookfield visc. 30 r.p.m., cp.*	*Penetration time sec.*
Ammonium polyacrylate, cross-linked[a]	4630	1.6
Ammonium polyacrylate, linear[a]	1920	1.9
Sodium polyacrylate	1660	1.7
Methylcellulose, 400 cp.	740	4.6
Methylcellulose, 4000 cp.	1420	4.2
Sodium carboxymethylcellulose	4900	4.5
Ammonium salt, carboxy vinyl polymer	2600	1.4
Sodium alginate, low viscosity	450	4.1
Gum arabic	30	1.4
Gum tragacanth	40	1.5
Gelatin, high bloom	390	<1.
Ammonium caseinate	490	1.5
Polyvinyl alcohol	2000	1.3
Bone glue	450	1.3

[a] Supplied as unneutralized emulsion.

Phase III

The third phase of this study consisted of a complete factorial experiment. The independent variables were:

Methylcellulose viscosity grade—3 levels: 400, 4000, 15,000 cp.
Methylcellulose concentration—2 levels: 0.1, 0.3 parts by weight per hundred parts clay
Per cent solids—3 levels: 50, 55, 60% total solids

The basic coating color used was as follows:

	Parts by weight, dry
Medium fraction coating grade clay	100
Latex	15
Modifier	0.1 or 0.3
Water	As required for indicated per cent solids

The results of the third phase of this investigation were analyzed as before, by means of a regression analysis, which yielded the least squares equations (5) and (6).

$$y_1 = 853.9 - 699.9\,x_1 + 145.2\,x_1^2 - 2141.\,x_2 + 1000.\,x_1x_2 \quad (5)$$

$$y_2 = 15.31 + 0.3734\,x_1^2 - 1.942\,x_1 + 17.35\,x_2 + 0.725\,x_3 - 0.00725\,x_3^2 \quad (6)$$

y_1 = coating color viscosity, cp.
y_2 = water retention, sec.
x_1 = log methylcellulose viscosity grade
x_2 = concentration of methylcellulose, parts per 100 parts clay
x_3 = per cent total solids

The variables studied had no significant effect on the pick resistance of paper coated with the colors used in phase III.

Referring to Fig. 10, it is seen that the largest single effect on water retention was the response to increasing the methylcellulose concentration. Referring to Fig. 9,

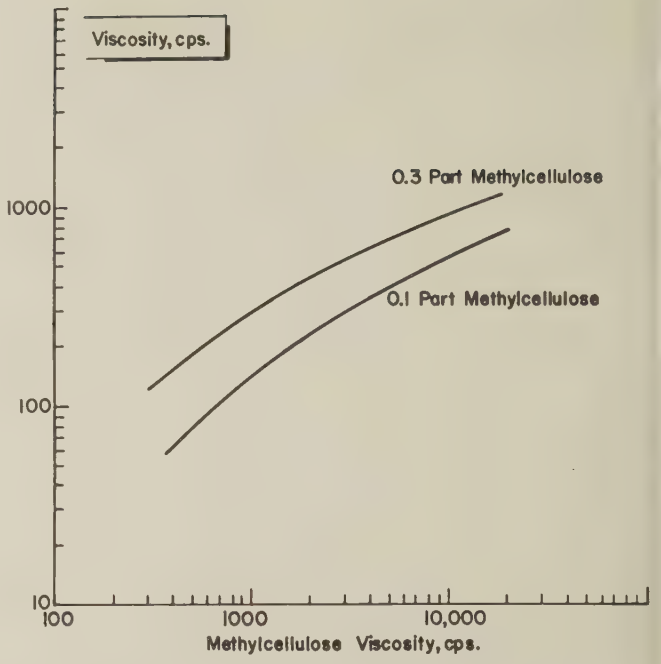

Fig. 9. Effect of methylcellulose viscosity on coating color viscosity

it is seen that the methylcellulose concentration also has a substantial effect on the viscosity of the coating color, although not so great as its effect on water retention. It is of interest that the per cent solids of the coating color has no significant effect on the viscosity, and only a slight effect on the water retention of the coating color. The curves shown in Fig. 10 also indicate that the water retention decreases with increasing per cent solids. No attempt is made to explain this phenomenon here, except to note that the same effect was observed in phase I when high proportions of latex were present in the adhesive.

In Fig. 11, the viscosities and penetration times determined from equations (5) and (6), respectively, are plotted against the viscosity grade of methylcellulose used as thickener. The values plotted for viscosity and penetration time are the values determined from

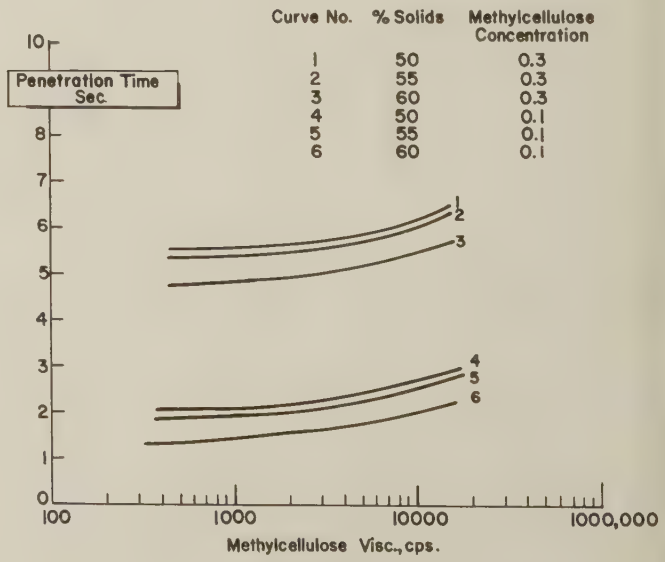

Fig. 10. Effect of methylcellulose viscosity on water retention

the equations divided by their respective standard deviations, in order to provide more nearly equivalent scales for plotting the two functions. It is clearly evident from Fig. 11 that there is very little correlation between viscosity and penetration time.

Table II. Effect of Order of Mixing

Point of addition of thickener	*Water retention, sec.*
1. None added	1.6
2. Added to clay slurry	9.1
3. Added to latex	8.8
4. Added to finished color	8.8

In order to determine whether the order of addition of the various ingredients in the basic formulation used for the third phase of this study had any effect on the water retention, a brief experiment was conducted. Using a formulation containing 0.5 parts methylcellulose (4000 cp. viscosity grade) as thickener at 50% total solids, the water retention values shown in Table II were obtained. It is apparent that the point at which the thickener was added to the coating color had no significant effect on the water retention.

SUMMARY

The manner in which several coating color variables affect the water retention of latex bound coating colors was determined. These effects are best described by the least squares equations determined in the body of the results. In addition to the variables whose effects were described in equation form, several synthetic thickeners were evaluated for their effectiveness in increasing the water retention of all-latex-bound coating colors. A few of the materials evaluated for this purpose seemed to show promise. Marked increases in water retention were obtained when the coating colors evaluated were thickened with one of the following: methylcellulose, carboxymethylcellulose, or sodium alginate.

Because of the effectiveness of the materials mentioned above in increasing water retention of latex bound coating colors, one of the materials, methylcellulose, was investigated in further detail. It was found that the concentration of methylcellulose present was much more important in increasing water retention than was the viscosity grade (molecular weight) of methylcellulose used. Methylcellulose was found to be as effective in increasing water retention of the latex bound colors studied as sodium alginate. It has been extensively claimed that the alginates are very effective in controlling the water retention of paper coating colors.

One of the basic differences between a synthetic latex and casein, i.e., the much greater hydrophobic character of latex than casein, was emphasized by the absence of correlation between water retention and pick resistance or viscosity. For example, with coatings containing high proportions of latex in the adhesive, the pick velocities obtained remained relatively high despite substantial variations in the corresponding water retention measurements. As the proportion of casein in the adhesive was increased, the pick results showed a progressively higher degree of correlation with water retention measurements. Previous workers had found a correlation between pick measurements and water retention using coating colors containing protein or starch adhesive.

Any conclusions to be made concerning adhesive redistribution in latex bound coating colors must await further experimentation. Such experimentation could consist of measurements of other properties and in addition would include other variables of interest such as the substrate, latex composition variables and latex-starch combinations. Since this investigation was intended to outline the importance of coating color variables and their effect on water retention, future investigations will consider the most important variables in greater detail.

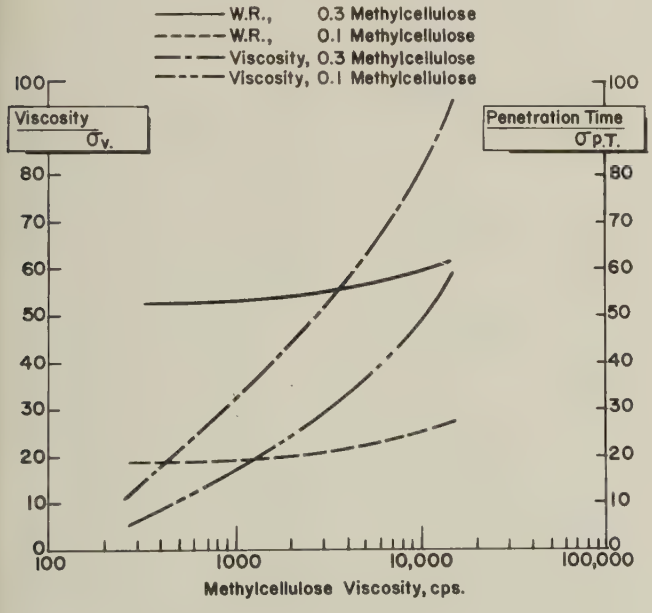

Fig. 11. Correlation of viscosity with water retention

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Suppliers of the materials used in this investigation were: Medium Fraction Coating Grade Clay—J. M. Huber Corp., New York City, N. Y., Minerals & Chemicals Corp. of America, McIntyre, Ga. Calcium Carbonate—Wyandotte Chemicals Corp., Wyandotte, Mich. Latex—The Dow Chemical Co., Midland, Mich. Titanium Dioxide—Titanium Pigment Corp., New York, N. Y., E. I. du Pont de Nemours & Co., Wilmington, Del. Casein—The Borden Chemical Co., Bainbridge, N. Y. Methylcellulose—The Dow Chemical Co., Midland, Mich.

The authors wish to express their appreciation to E. C. Yehle, The Dow Chemical Co., Midland, Mich., for his assistance in the statistical work involved in this investigation.

A Comparison of the Carbohydrates of Hardwoods and Softwoods

J. K. HAMILTON and N. S. THOMPSON

Holocelluloses have been prepared from a number of hardwoods and softwoods by the chlorite method and, in certain instances, by a new milder method employing chlorine dioxide buffered to pH 7. Western red alder has been shown to contain a glucomannan with a glucose-to-mannose ratio of 2:3. The isolation and identification of crystalline *O*-α-4-*O*-methylglucuronopyranosyl-(1 → 2)-*O*-β-D-xylopyranosyl-(1 → 4)-D-xylopyranose-trihydrate indicates that the 4-*O*-methylglucurono-xylan of this wood is similar to that from other hardwoods. The results of pretreatment and high-strength sodium hydroxide extractions of various holocelluloses indicate that most of the hemicelluloses are not chemically combined with the cellulose of either type of wood. The above studies, in addition to work reported previously from this laboratory and the general literature on the subject, point to several differences between the noncellulosic polysaccharides found in hardwoods and softwoods. The cellulose of these two types of wood is very similar in physical and chemical properties. Hardwoods contain about 20 to 30% hemicelluloses, the main constituent being a 4-*O*-methylglucurono-xylan with a high ratio of D-xylose to 4-*O*-methyl-D-glucuronic acid. These woods also contain small amounts of mannose, galactose, and arabinose. Most of the mannose appears to be associated with a highly inaccessible glucomannan. Softwoods contain about 15 to 20% hemicelluloses, the main constituent being a difficultly extractable glucomannan. Smaller amounts of a 4-*O*-methylglucurono-araboxylan, which has a lower ratio of D-xylose to 4-*O*-methyl-D-glucuronic acid, also occur. Soluble glucomannans, galactoglucomannans, and arabogalactans occur to a lesser but significant extent. Pectic material, starch, traces of rhamnose and glucuronic acid occur in both types of wood.

THE economic importance of hardwoods has greatly increased in recent years. The main reasons for this increase are probably (1) a more efficient use of timber resources, (2) the development of new pulping techniques to handle hardwoods more efficiently, and (3) the fact that some hardwoods contain more cellulose than softwoods. It is interesting and highly pertinent, therefore, to examine the carbohydrate constituents of hardwoods and to compare them with the lesser known and more complex carbohydrate polymers of the heretofore much more economically important softwoods.

The predominant carbohydrate polymer in either softwoods or hardwoods is cellulose, and, irrespective of where it is isolated, it is very similar in chemical and physical properties. A comparison of the cellulose of hardwoods, softwoods and grasses is currently receiving intensive examination by Timell (*1*) and Snyder and Timell (*2*).

The hemicelluloses rank second to cellulose in abundance in nature and are mainly associated with the cell wall, although a minor source of carbohydrate material, which appears to be a mixture of pectic substances and hemicelluloses, occurs in the middle lamella. Lignin, which also exists mainly in the middle lamella, occurs to a lesser extent in hardwoods (20 to 25%) than in softwoods (27 to 31%) and has a higher methoxyl content in the former (20 to 22%) than in the latter (14 to 16%). The extractives, the amount and nature of which differ markedly in both groups, are often an important factor in determining the ultimate properties and end uses of the wood.

Hardwoods contain on the average 20 to 30% hemicelluloses, the main constituent being D-xylose; whereas the softwoods contain 15 to 20% hemicelluloses, the main constituent being D-mannose, with smaller amounts of D-yxlose. Because of the purer nature of the hardwood hemicellulose and because of the ease of isolating it from the wood, the hardwood hemicelluloses have been the subject of many more investigations over a longer period of time than the softwoods. The fact that hardwood hemicelluloses contain a high proportion of D-xylose and also contain a methyl hexuronic acid (now known to be 4-*O*-methyl-D-glucuronic acid) has been known since the classical studies of O'Dwyer (*3*) and Sands (*4*).

In recent years the use of paper partition chromatography for the separation of various mono- and oligosaccharides has enabled investigators to examine the hydrolyzates of carbohydrate polymers much more critically than has been possible in the past. This procedure has resulted in much more accurate analyses and, coupled with ion exchange resins, has resulted in the detection and isolation of many important carbohydrate oligosaccharides and oligouronides. The modern techniques for the fractionation of polymeric materials, coupled with chromatographic examination of the hydrolyzed fractions, has resulted in the detection, isolation, and purification of many new polymeric systems of wood such as 4-*O*-methyl-D-glucurono-araboxylans, glucomannans, and galactoglucomannans.

At the present time, although quite a number of different carbohydrate polymers have been isolated, purified, and characterized, very little is known about the absolute amount in the wood fiber. Even less is known about the location of these polymers in the wood fiber. Certain generalizations may be made, however, concerning the quantity and type of polymer present in the different woods.

This paper is concerned with a comparison of the carbohydrate material associated with both hardwoods and softwoods. It will deal mainly with the hemicelluloses and, in an attempt to unify the general observations recorded in the literature, the results of simple experiments carried out in this laboratory will be inter-

J. K. HAMILTON and N. S. THOMPSON, Olympic Research Div., Rayonier Inc., Shelton, Wash.

woven throughout. These small-scale experiments represent a survey which was carried out in order to find efficient methods for the separation of the various hemicelluloses of hardwoods. A similar survey is currently under way on the hemicelluloses of softwoods. In general, this survey has been successful and, in certain instances, large-scale extractions have been carried out, and the hemicelluloses are being characterized by conventional chemical procedures.

EXPERIMENTAL

Analytical Procedures

The solubilities of the pulps and holocelluloses in 10 and 18% NaOH (S_{10} and S_{18}) are quoted in the tables as they are more readily obtained and are more meaningful than the usual alpha, beta, and gamma-cellulose contents. The significance and use of these measurements have been described by Goldschmid and Jeffery (*5*).

The analyses of the pulps and their extracts have been corrected for their moisture and ash contents. Optical rotations were determined on a 1% solution of the polymers in 10% NaOH.

The pulps were hydrolyzed by solution in 72% H_2SO_4 followed by dilution (to 4%) and heating under reflux for 8 hr. The hydrolyzates, after isolation and the appropriate dilution, were chromatographed on suitable solvents and analyzed for individual sugars by a quantitative paper chromatographic method similar to that of McCready and McComb (*6*). The chromatographic solvents employed in this investigation were described in greater detail in a previous communication (*7*). One may preferentially separate oligosaccharides, oligouronides, neutral sugars, or uronic acids according to the solvent employed. A solvent system composed of butyl acetate-pyridine, -ethanol (95%)-water 8:2:2:1 v./v. (*32*) was found most satisfactory for separating galactose, glucose, mannose, arabinose, and xylose for quantitative estimations.

Acid-insoluble lignin was determined by the TAPPI procedure (*8*), while the soluble lignin in the holocelluloses was measured by the spectrophotometric method of Browning and Bublitz (*9*). The cuene I.V. was determined by measuring the viscosity of the hemicelluloses in molar cupriethylenediamine hydroxide and is expressed in deciliters per gram.

Holocellulose and Pulp Preparation

The holocelluloses employed in this investigation were prepared by two techniques. One method employed a modification of the procedures originally published by Jayme (*10*), Lovell (*11*), and Wise (*12*). In this case the woods were chipped to pass a $^1/_2$-in. screen and screened on a $^1/_{16}$-in. screen to remove fines, leaving only medium-sized chips. The large chips were removed by hand. Sodium chlorite (6% pH 4.5) was employed at a temperature of 47 ± 2°C. (except the southern pine blend, 63 ± 2°C.) for periods of 50 to 100 hr. The solution was changed four or five times during this period. The longer times were required by the hemlock, alder, and gumwood. The holocellulose was mechanically disintegrated, washed thoroughly with water, and dried slowly by solvent exchange, using several changes of methanol and acetone.

The other holocellulose method employed, as an oxidizing medium, an aqueous solution saturated with respect to chlorine dioxide and sodium bicarbonate. This solution (pH 7.5–8.0, 1 l. for each 150 g. of air dry wood) was added to thin, radial-section, degassed chips of known moisture content. The chips averaged 5 cm. in length, 1 to 2 cm. in width, and 1 mm. in thickness. All oxidations were carried out at room temperature (23 to 25°C.). Thin chips, rather than the more widely used wood meal, were employed since it was desired to isolate whole fibers. It was felt that the highly soluble hemicelluloses located in the relatively inaccessible portions of the fiber would not be so readily extracted by the aqueous oxidizing solution. When most of the chlorine dioxide had been consumed, which occurred after 1 to 3 hr. (depending upon the state of delignification of the sample), the chips were filtered and reoxidized. After four oxidations, the chips were extracted for 1 week with methanol (solvent changed daily). It was found that individual oxidations should not exceed 4 hr. as the pH became greater than 8 as the sodium bicarbonate was converted to the salts of weak organic acids.

The chips darkened during the methanol extraction period, presumably because of the diffusion of partly oxidized lignin to the surface of the chips or perhaps because of the removal of oxidized lignin leaving a surface of partly oxidized ligneous material. After 1 week, the chips were reoxidized four times as before. Generally, about eight to 12 oxidation and extraction cycles were required, the number being dependent upon the type of wood and chip size. During the later stages, the wood chips were osterized briefly in methanol to break up the larger fiber bundles and to assist in the penetration of the oxidant. Care was also taken during the later stages to remove fully bleached fibers and fiber bundles from the bulk of the material. Resistant fragments of resinous wood were also removed. These fractions could then be oxidized separately according to their conditions without too much danger of overoxidation or overextraction.

The carbohydrate material which dissolved in the chlorine dioxide-sodium bicarbonate oxidizing solution was isolated following dialysis, evaporation under diminished pressure and precipitation with methanol. This fraction amounted to 3.9% for western red alder and about 2% for madrona. Analysis of this fraction from western alder gave 9.3% ash, 10% moisture, 19.5% Klason lignin, 3.4% soluble lignin, 2.8% galactose, 1.5% glucose, 0.9% mannose, 2.1% arabinose, 6.2% xylose, and a trace of rhamnose. Since strong chromatographic spots were obtained corresponding to galacturonic acid (only a questionable trace of glucurone was noted), it was presumed that the remainder (44.3%) was pectic acid.

The neutral sulphite semichemical pulp was prepared by the conditions described by Husband for a high-yield pulp (*13*). This pulp was bleached by conventional techniques. The analytical properties of western red alder (*Alnus rubra*), prepared by all three processes, and various other deciduous and coniferous woods are given in Tables I and II.

Extraction of Hardwood Chlorine Dioxide Holocelluloses

The alder holocellulose was extracted with 0.1 *N*

Table I. Analytical Data for Acid Chlorite Holocelluloses of Softwoods and Hardwoods

	Western hemlock[a]	*Southern pine*[b]	*Western red alder*[c]	*Gumwood*[d]
Yield,[e] %	65.3	62	68.5	71
Soluble lignin of holocellulose, %	4.2	3.9	3.6	4.6
Insoluble lignin of holocellulose, %	0.2	0.2	0.04	0.2
Yield on holocellulose,[e] %	60.9	57.9	64.9	66.2
Cuene I.V., dl./g.	8.1	7.5	6.1	6.8
S_{10}, %	25.9	23.8	25.3	26.0
S_{18}, %	23.1	18.9	22.4	19.4
Galactose, %	1.3	1.1	0.9	0.9
Mannose, %	14.4	11.1	0.7	1.9
Arabinose, %	0.7	0.7	0.1	0.3
Xylose, %	2.9	6.8	19.2	16.6
4-*O*-methyl-D-glucuronic acid[f]	Present	Present	Present	Present
Cellulose,[g] %	41.7	44.3	45.0	46.5

[a] *Tsuga heterophylla.*
[b] An equal mixture of *Pinus elliottii* var. *elliottii* and *Pinus palustris.*
[c] *Alnus rubra.*
[d] A 3:2:1 blend of red gum (*Liquidambar styraciflua*):tupelo gum (*Nyssa aquatica*):black gum (*Nyssa sylvatica* var. *biflora*).
[e] Oven-dry yield based on oven-dry wood.
[f] 4-*O*-methyl-D-glucuronic acid detected by paper partition chromatography; approximate content 10% of xylose for hardwoods and 15% of xylose for softwoods.
[g] Calculated from glucose (by difference) and corrected for glucose present in glucomannans and galactoglucomannans.

NaOH, and it was found that the fiber bundles disintegrated completely. Sodium carbonate (5%, about pH 11) was not able to disintegrate all the fiber bundles. The extract from the alder fibers amounted to 2.3% and was composed of uronic acids, galactose, glucose, mannose, arabinose, xylose and traces of rhamnose as

Table II. Analytical Data for Chlorine Dioxide and Neutral Sulphite Semichemical Hardwood Holocelluloses and Pulps

Wood →	*Western red alder*[a]		*Madrona*[b]
Method of preparation →	ClO_2	*Neutral sulphite semi-chemical*	ClO_2
Yield (on wood),[c] %	78.9	74[d]	71.2[e]
Soluble lignin of holocellulose,[c] %	2.6	[f]	2.1
Insoluble lignin of holocellulose,[c] %	3.2	[f]	2.9
Soluble extra-cellular carbohydrates,[c,i] %	1.5	[f]	[f]
Holocellulose yield (on wood),[c] %	73.1[k]	[f]	66.2
Cuene I.V., dl./g.	10.0	8.0	[f]
Galactose,[g] %	0.6	0	0.8
Glucose,[g] %	70.0	77.3[h]	59.3
Mannose,[g] %	1.8	1.0	2.3
Arabinose,[g] %	0.1	0	0.6
Xylose,[g] %	27.5	21.7	37.0
Rhamnose,[g] %	Trace	0	Trace
4-*O*-methyl-D-glucuronic acid [g,i]	Present	Present	Present

[a] *Alnus rubra.*
[b] *Arbutus menzeii.*
[c] Calculated on oven-dry wood.
[d] Unbleached pulp yield.
[e] Serious mechanical losses.
[f] Not measured.
[g] Based on holocellulose.
[h] By difference.
[i] 4-*O*-methyl-D-glucuronic acid determined by paper partition chromatography.
[j] Corrected for moisture, lignin, and ash.
[k] After extraction with 0.1N NaOH.

determined by qualitative paper chromatography. This extraction removed most of the galactose from the holocellulose.

A similar sample of alder holocellulose was Wiley milled to pass a 40-mesh screen and was also extracted with 0.1 *N* NaOH under identical conditions. Qualitative examination of the extract (5.2% of the pulp) showed the same sugars were present as above but with much larger amounts of xylose and 4-*O*-methyl-D-glucuronic acid. Quantitative examination of the extracted holocellulose showed very slightly smaller amounts of galactose, arabinose, and mannose to be

Table III. The 4-*O*-methylglucurono-xylans Extracted from Hardwoods with 10% KOH

Wood →	*Western red alder*			
Pulping procedure →	ClO_2	*Neutral sulphite semi-chemical*	$NaClO_2$[a]	*Madrona* ClO_2
Yield, %	24.3	20.2	14.5	21.8
$[\alpha]_D^{25}$	−70.5°	−70.6°	−64°	−72°
OCH_3, %	[b]	2.1	2.2	2.5
Cuene I.V., dl./g.	0.67	0.66	0.55	0.80
Molar ratio of sugars[c]				
Glucose	Trace	?	Trace[a]	Trace
Xylose	Large	10	9	8
4-*O*-methyl-D-glucuronic acid[d]	Present	1	1	1

[a] This holocellulose was extracted with 5% NaOH and not 10% KOH. In addition, it was not pre-extracted with 0.1 *N* NaOH, and the extract therefore contained small amounts of glucose, mannose, galactose and arabinose in addition to xylose and uronic acid in a 0.2:0.3:0.2:0.1:9:1 molar ratio. This polymer has been described elsewhere (*18*).
[b] Not analyzed.
[c] Examination of numerous similar extracts has shown the inconsistent appearance of arabinose in some hydrolyzates.
[d] The 4-*O*-methyl-D-glucuronic acid was qualitatively identified by paper chromatography and quantitatively measured by the methoxyl content of the polymer.

present, while the loss of xylose corresponded to the 3% gain in yield of the latter extract.

Samples of madrona holocellulose, Wiley milled and extracted under similar conditions, gave an extract containing the same sugars listed above but in slightly different amounts. The 0.1 *N* NaOH extracts of alder, madrona, gumwood, and elm (*Ulmus pumila*) holocellulose fibers gave strong positive iodine tests for starch.

The madrona and alder chlorine dioxide holocelluloses (previously extracted with 0.1 *N* NaOH) were next extracted with 10% KOH (10% consistency). The polymers isolated proved to be 4-*O*-methylglucuronoxylans as indicated by qualitative chromatography of graded hydrolysis products. The analytical characteristics of some of these polysaccharides are given in Table III. Exceedingly small amounts of glucose and, inconsistently, traces of arabinose were detected in these extracts.

The madrona and alder chlorine dioxide holocelluloses which had been extracted with 10% KOH were next extracted with 10% NaOH (10% consistency). The analyses of the extracts are given in Table IV. Of particular interest is the more positive rotation and

Table IV. The 10% NaOH Extracts of Chlorine Dioxide Hardwood Holocelluloses

	Western red alder	*Madrona*
Yield, %	5.5	6.6
Cuene I.V., dl./g.	0.41	0.27
$[\alpha]_D^{25}$	−20.5°	−9.7°
OCH_3, %	0.93	0.96
Molar ratio of sugars		
Glucose	7	3
Mannose	5	3
Xylose	9	17
4-*O*-methyl-D-glucuronic acid[a]	1	1

[a] The 4-*O*-methyl-D-glucuronic acid was qualitatively identified by paper chromatography and quantitatively measured by the methoxyl content of the polymer.

much smaller cuene I.V. of these fractions. The major sugars produced by hydrolysis were glucose, mannose, and xylose in the ratios shown in Table IV, while trace amounts of galactose and arabinose were detected. These samples were fractionated by a variety of techniques and separated into mannose, xylose, and glucose-rich fractions. The most successful procedure will be described below.

Table V. The Fractionation of the 10% NaOH Extract of Neutral Sulphite Semichemical Pulp of Western Red Alder

Alkali used →	10% NaOH soluble					
Fraction on acidification →	Insoluble			Soluble		
Behavior on extraction with 10% KOH →	*Ins. in 10% KOH*	Soluble		*Ins. in 10% KOH*	Soluble	
Fraction on acidification →		*Insol.*	*Sol.*		*Insol.*	*Sol.*
Yield on pulp, %	0.75	0.42	0.28	0.42	0.35	0.20
Molar ratio of sugars						
Galactose	0	0	1.0	0	0	1.0
Glucose	6.6	0	3.9	1.0	0	3.0
Mannose	1.7	0	2.3	1.7	0	2.0
Arabinose	0	0	0	0	0	0.6
Xylose	0.8	L	14.8	0.7	L	8.5
4-*O*-methyl-D-glucuronic acid	P	P	P	P	P	P
Fraction no.	A	B	C	D	E	F

L = large. P = present.

The Extraction of Neutral Sulphite Semichemical Alder Pulp

Three hundred grams of a neutral semichemical pulp similar to that described in Table II was extracted four times with 10% KOH (10% consistency). While still wet, the pulp was extracted four times with 10% NaOH (10% consistency). The 10% KOH extract of the pulp was neutralized with acetic acid, evaporated until potassium acetate began to crystallize, was flooded with methanol, and the 4-*O*-methylglucurono-xylan isolated. No appreciable glucose was associated with this polymer. The properties of this polysaccharide are shown in Table III and are quite similar to the original polymer extracted from the holocellulose. A sample of the 4-*O*-methylglucurono-xylan (20 g.) was subjected to a graded hydrolysis in 0.25 *N* H_2SO_4 according to the procedure described elsewhere (*7*). Oligouronides were isolated using paper chromatographic separation on Whatman no. 17 paper with a Whatman no. 1 wick in the amounts: 4-*O*-methyl-D-glucuronic acid, 0.6%; 2-*O*-(4-*O*-methyl-α-D-glucuronopyranosyl)-D-xylose, 1.7%; *O*-α-4-*O*-methyl-D-glucuronopyranosyl-(1 → 2)-*O*-β-D-xylopyranose-(1 → 4)-D-xylopyranose, 2.7%; and an aldotetraouronic acid, 0.6%. These oligouronides were identified by paper chromatography on several solvents by comparison with authentic specimens. The aldotriouronic acid from alder crystallized under the typical conditions necessary for the crystallization of authentic *O*-α-4-*O*-methyl-D-glucuronopyranosyl-(1 → 2)-*O*-β-D-xylopyranosyl-(1 → 4)-D-xylopyranose trihydrate. It was further identified by comparison with an authentic specimen from western hemlock by the decomposition characteristics upon heating, optical rotation (+ 56° versus + 57°), equivalent weight (531 versus 526), methoxyl content (6.1 versus 5.9%) and the identity of the two infrared absorption spectra.

The 10% NaOH extract of the alder pulp was neutralized with acetic acid and separated into soluble and insoluble fractions. Each fraction was washed free of residual salt with methanol and was then (while still wet) back-extracted with 10% KOH. A portion of each fraction which did not dissolve was neutralized and washed free of salt with methanol, then ether and dried. The two fractions soluble in 10% KOH were neutralized with acetic acid and were divided into soluble and insoluble fractions. A total of six fractions were isolated and are described in Table V. They are labeled A, B, C, D, E, and F for convenience in the text.

Those fractions which did not dissolve in 10% KOH (A and D) were rich in glucose and mannose. Those which did dissolve (B and E) but precipitated upon neutralization were 4-*O*-methylglucurono-xylans, while the soluble fractions (C and F) upon hydrolysis contained galactose, glucose, mannose, xylose, 4-*O*-methyl-D-glucuronic acid and, in one instance (F), arabinose. The two fractions richest in mannose (A and D) were dissolved in 5% NaOH and fractionated by the gradual addition of saturated aqueous barium hydroxide (*14, 75*). The most successful fractionation was accomplished by dissolving 0.913 g. of the crude fraction D in 100 ml. of 5% NaOH. Saturated aqueous barium hydroxide was added in the manner shown in Table VI. A total of five fractions was obtained, three of which appeared to be relatively pure glucomannans. A small amount of this glucomannan (30 mg.), together with controls consisting of glucomannans from western hemlock and southern pine, was subjected to a standard graded hydrolysis (*14*). Chromatographic examination of the hydrolyzate showed substances which corresponded to the mannobiose, glucosidomannose, mannosidoglucose, and cellobiose from the softwood glucomannan standards.

Fraction C was also fractionated from 10% NaOH by the addition of saturated barium hydroxide. Three fractions were obtained, the first of which was rich in glucose and mannose (with a small amount of xylose), the second was rich in galactose, glucose, mannose, and xylose and the third was rich in galactose, glucose, and xylose.

Table VI. The Fraction of Glucomannan-Rich Fraction D of Western Red Alder

Fraction	*Cumulative vol. of $Ba(OH)_2$*[a]	*Per cent of fraction precipitated*	*Molar ratios of sugar*			$[\alpha]_D^{25}$	*Cuene I. V.*[b]
			Glucose	*Mannose*	*Xylose*		
1	20	24	1.0	1.58	0.02		
2	40	12	1.0	1.51	0.07	−32°[c]	0.24
3	60	8	1.0	1.41	0.07		
4	80	2	1.0	0.9	1.0	...	...
5	Soluble	34	1.0	1.1	4.1	−20.4°	0.33

[a] Saturated solution.
[b] dl./g.
[c] Fractions 1, 2, and 3 were combined.

Successive Extraction of Holocellulose with Sodium Hydroxide of Varying Strength

Hardwood and softwood chlorite holocelluloses (see Table I) were employed for another series of extraction experiments. Although these holocelluloses had been prepared at a lower temperature than is usual in preparing chlorite holocellulose, a slight loss of carbohydrate material occurred. This small loss was not considered a drawback in these experiments. The holocelluloses were given an initial 5-min., high-temperature (98°C.) hydrolysis and were then subjected to three extractions with 10% NaOH followed by three extractions with 18% NaOH (all at 25°C.) to remove as much hemicellulosic material as possible. The extracted residue of each holocellulose was analyzed for yield, intrinsic viscosity, and the percentage xylose and mannose. More specific details of this procedure were published elsewhere (*15*). Table VII contains the results of these experiments on western hemlock (*Tsuga heterophylla*), western red alder (*Alnus rubra*), southern pine (a mixture of *Pinus elliottii* var. *elliottii* and *Pinus palustris*), and gumwood (predominantly *Liquidambar styraciflua* —see Table I).

Table VII. A Comparison of Certain Chemical Properties of Sodium Hydroxide-extracted Acid Chlorite Holocelluloses[a]

	Western hemlock	*Southern pine blend*	*Western red alder*	*Gumwood blend*
Yield (on wood), %	32	28.2	28.2	30.2
Cuene I.V., dl./g.[b]	8.1	...	...	6.2
Cuene I.V., dl./g.[c]	8.7	...	...	8.0
Cuene I.V., dl./g.[d]	7.2[e]	...	...	5.4
Arabinose, %	0	0	0	0
Galactose, %	0	0	0	0
Xylose, %	0.03	0.02	0.03	0.03
Mannose, %	0.18	0.19	0.03	0.05
4-*O*-methyl-D-glucuronic acid	0	0	0	0

[a] All values, where applicable, are based on percentage of oven-dry wood. (See Table I for identity of woods.)
[b] Value prior to extraction.
[c] Wiley milled and extracted.
[d] Mild acid hydrolysis, Wiley milled and extracted.
[e] Estimated value.

RESULTS

The carbohydrate analyses of various hardwood and softwood holocelluloses prepared by the chlorite method are given in Table I. For comparison, Table II shows the analyses of hardwood chlorine dioxide and neutral sulphite semichemical pulps. A comparison of the western red alder pulps prepared by all three processes shows that the chlorine dioxide technique appears to be the mildest.

The chlorine dioxide holocellulose procedure has given an additional carbohydrate fraction which, by qualitative examination, appeared to be a mixture of a polygalacturonic acid and an unknown carbohydrate system containing galactose, glucose, arabinose, xylose, rhamnose, and possibly glucuronic acid. These same fractions appeared to be present in the hydrolyzate of the holocelluloses, and attempts were made to extract them from the alder holocellulose with mild extraction agents. Sodium carbonate (5%) was not particularly effective in breaking up the fiber bundles of holocellulose or in extracting any material. Tenth normal sodium hydroxide was more effective, and the fiber bundles collapsed with a little stirring (*76*). The extract amounted to 2.3% and was composed of galactose, glucose, mannose, arabinose, xylose, uronic acids, and traces of rhamnose. Strong positive iodine tests indicated that much of the glucose was present as starch. Samples of chlorine dioxide western red alder and madrona holocelluloses were Wiley milled to pass a 40-mesh screen and were extracted with 0.1 *N* NaOH. In the case of alder, the extract amounted to 5.2% of the holocellulose and was composed of the same sugars mentioned above but with larger amounts of xylose. The increased yield appears to have been the result of the extraction of 4-*O*-methylglucurono-xylan made available by breaking the fiber structure. The fractions which dissolved in the oxidizing solution and in 0.1 *N* NaOH from whole fibers of hardwood holocellulose were assumed to be "extra-cellular" hemicelluloses because of their apparent release as delignification proceeds and because of the ease with which they are dissolved in alkali from whole fibers and fiber bundles.

The two chlorine dioxide holocelluloses (alder and madrona) which had been Wiley milled and extracted with 0.1 *N* NaOH were next extracted with 10% KOH (see Table III). The extracts of both hardwoods had properties which were identical to 4-*O*-methylglucurono-xylans and resembled those extracted from chlorite and neutral sulphite semichemical alder pulps very closely. These polymers from alder and madrona were subjected to a graded acid hydrolysis and gave the expected oligosaccharides and oligouronides as determined by qualitative paper chromatographic comparison with the products obtained from the 4-*O*-methylglucurono-xylan of western hemlock sulphite pulp (*7*). These results lend support to the view that the 4-*O*-methylglucurono-xylans of alder and madrona do not differ in any major respect from the corresponding polymers isolated from other hardwoods.

The pulp mats of alder and madrona holocelluloses were next extracted with 10% NaOH (see Table IV) and the hemicelluloses isolated as before. The high yield of glucose and the higher optical rotation of $-10°$ to $-20°$ indicate that this material was chiefly a short chain glucan. The xylose probably arose from trace quantities of resistant 4-*O*-methylglucurono-xylan or related polymers. The mannose, as will be seen later, was derived from a glucomannan, the presence of which, in poplar, was demonstrated by Merler, Wise, and Jones (*16*). The extracts of alder and madrona were quite similar and probably indicates that both woods contain similar compounds although in different amounts.

In order to have sufficient glucomannan for this preliminary study, 300 g. of neutral sulphite semichemical alder pulp was extracted, first with 10% KOH to remove the 4-*O*-methylglucurono-xylan and then with 10% NaOH (see Table V) to remove the glucomannan. The 4-*O*-methylglucurono-xylan was examined quantitatively by graded acid hydrolysis. The proof of the identity of crystalline *O*-α-4-*O*-methyl-D-glucurono-pyranosyl-(1 → 2)-*O*-β-D-xylopyranosyl-(1 → 4)-D-xylopyranose trihydrate (which represents the branch point of the polymer) verifies the conjectured structure given above. The 10% NaOH extract (Table V) was neutralized and divided into soluble and insoluble fractions. Both were back extracted with 10% KOH, yielding

insoluble fractions A and D (Table V). The soluble fractions were divided into those soluble and insoluble upon neutralization, yielding fractions B, C, E, and F. Fractions A and D contained mannose in the form of a glucomannan. Fractions B and E, which were insoluble upon neutralization, were relatively pure 4-*O*-methylglucurono-xylans and were not examined further. Fractions C and F contain galactose polymers in addition to pentose-containing polymers. Fractionation of an alkaline solution of fraction C with successive additions of small volumes of saturated aqueous barium hydroxide indicated that this fraction was composed of a glucomannan, a 4-*O*-methylglucurono-xylan, and a fraction which was composed of polymers of glucose and galactose.

Fractions A and D were redissolved in 10% NaOH and were fractionally precipitated by the gradual addition of saturated aqueous barium hydroxide (see Table VI). Fraction A was found to be a complex mixture of a glucan and a glucomannan. Fraction D yielded some 44% as a glucomannan which had a 2:3 glucose-to-mannose ratio and an optical rotation of −32°. The corresponding fraction from western hemlock had a 1:3 ratio and a rotation of −37°.

The results of the successive extractions of the softwood and hardwood holocelluloses are shown in Table VII. This work represents an extension of previous work (*15*) on softwoods to hardwoods. The tables show that most of the hemicelluloses of hardwoods and softwoods may be removed from the cellulose portion of the fiber.

DISCUSSION

The study of the carbohydrate systems in wood is complicated by the presence of lignin and, in general, the hemicelluloses are not readily extracted from ligneous tissues. The extraction of finely divided hardwood usually gives a higher yield of carbohydrate material than does a similar extraction of a softwood (*17*). In those cases where such an extraction is successful, it is usually necessary to carry out a very tedious purification process.

Delignification permits very easy extraction of hemicelluloses from the residual wood, but the delignification process often results in the loss of varying amounts of carbohydrate polymers and usually results in the hydrolytic degradation of the residual polymers. Delignification procedures range from the commercial techniques such as sulphite or kraft cooking to the mildest laboratory method. In addition to removing hemicelluloses and to decreasing the D.P. of the residual polymers, certain processes may completely change the nature of the hemicelluloses in the resulting pulps. Examples are the conversion of 4-*O*-methylglucurono-araboxylan to 4-*O*-methylglucurono-xylan by the sulphite cook and the conversion of the same polymer to an araboxylan by the conventional kraft cook and to a xylan by a prehydrolyzed kraft cook (*18*). These "artifact" hemicelluloses are partly responsible for the great difference between pulps prepared by alkaline, neutral, and acidic pulping conditions.

The milder delignifications employed in the laboratory do not generally bring about such changes, although the loss of arabinose units from arabogalactans and 4-*O*-methylglucurono-araboxylans can occur at higher temperatures and hydrogen ion concentrations. The chlorite procedure normally carried out at pH 4 to 4.5 devised by Jayme (*10*) and modified by Wise (*12*), Lovell (*11*), and others is known to degrade carbohydrates and to extract considerable hemicellulosic material (*19–25*). The delignification procedures developed by Thomas (*26*), Timell and Jahn (*27*), and others appear to be reasonably suitable methods for the preparation of holocellulose from wood. These latter techniques all employ the oxidative action of chlorine upon moist wood (with different degrees of refinement) followed by extraction with ethanolamine in ethanol or some similar type of solvent system.

In this laboratory, work has been directed to a study of the behavior of hemicelluloses during various pulping procedures. It became of interest to isolate all of the cellular carbohydrates of wood, separately if possible, from the extra-cellular carbohydrates. The chlorite method was tried but was found wanting for the reasons mentioned previously. The chlorine method of Thomas (*26*) and Timell and Jahn (*27*) was not tried, although with suitable modifications it might have been successful. Instead, the chlorine dioxide technique originally developed by Schmidt and co-workers (*28*) was adapted to take advantage of the relatively high oxidation potential of the oxide in neutral solution (*29, 30*), its specificity for lignin and the solubility of the oxidized lignins in methanol (*31*).

Thin radial chips were oxidized in order to preserve as much fiber structure as possible. The oxidation liquors were dialyzed, and the dissolved carbohydrates were isolated. The chips were exhaustively extracted with methanol. Tests showed no carbohydrate material was present in the yellow methanol solutions of degraded lignin. These oxidation and extraction cycles were repeated until a suitable degree of delignification was achieved.

Tables I and II show that the chlorine dioxide-sodium bicarbonate method of preparing holocelluloses appears to give a more satisfactory holocellulose than the chlorite method or the semichemical pulping procedure. The method appears to be equally applicable to the preparation of coniferous holocellulose according to preliminary data obtained during the delignification of *Tsuga heterophylla* (*32*). The carbohydrate content of *Alnus rubra* prepared by this method resembles that of *Alnus incanna* (*33*) very closely, but both alder samples had less hemicellulose (i.e., 4-*O*-methylglucurono-xylan) than *Betula papyrifera* (*34*) and *Betula verrucosa* (*33*), other common members of the Betulaceae family.

Some 3.9% of the hardwood carbohydrates dissolved in the oxidizing medium and seemed to be the same as the 2.3% which could be dissolved from whole fibers and fiber bundles (with destruction of the fiber bundles) with 0.1 *N* NaOH. This fraction (as inferred by qualitative paper chromatography of the hydrolyzates) consisted of a complex mixture of polygalacturonic acid and a polymeric mixture composed of galactose, arabinose, xylose, glucose (most likely as starch), mannose, rhamnose, and possibly glucuronic acid. The nature of these polymers of hardwood has not been completely elucidated at present but they have been detected by other workers (*26, 76, 77*). Polygalacturonic acid has been detected in black locust (*Robinia pseudoacacia*,

L.) (*35*), lemon wood (*Citrus limonia*, Obek), and mesquite wood (*Prosopis juliflora*) by Anderson and co-workers (*36*). Timel and co-workers have found galacturonic acid in the hydrolyzates of white birch (*37*) and white elm (*38*) holocelluloses. Other evidence for the presence of this complex mixture in hardwoods has been found in poplar (*16*) and gumwood (*18*). The trace amounts of polymers containing galactose, mannose, and arabinose, which remain after extraction with 0.1 *N* NaOH, are not extracted with 5% NaOH or 10% KOH but may be partly removed with 10% NaOH. It would appear, therefore, that these two new fractions are different and represent accessible and quite inaccessible, but nevertheless soluble, polymers.

The fraction of softwoods which corresponds to this "pectic" fraction from hardwoods has received considerably greater attention. Pectic acid has been detected in white pine (*Pinus strobus* L.) by Anderson and co-workers (*36*), and the isolation, identification, and characterization of arabogalactans from larch (*Larix occidentallis*) (*39–42*), (Larix decidua) *43–44*, Jeffrey pine (*Pinus jeffreyi*) (*46*), jack pine (*Pinus banksiana*) (*45*), longleaf pine (*Pinus palustris*) (*47*), white spruce (*Picea glauca*) (*48*), and black spruce (*Picea mariana*) (*49*) has been reported. Preliminary work on the chlorine dioxide-sodium bicarbonate soluble carbohydrates of western hemlock (*Tsuga heterophylla*) indicates that these polymers also constitute the "pectic" or "extracellular" carbohydrates of that species (*32*).

Another highly soluble polymer recently found in softwoods is the branched galactoglucomannan (*32*). This polymer was extracted from finely ground spruce wood (*Picea glauca*) by Adams (*48*), and has also been detected in conventional kraft hemlock, soda hemlock, hemlock holocellulose, conventional kraft southern pine, southern pine holocellulose, conventional kraft redwood, and soda redwood pulps (*18*). The galactoglucomannan of southern pine has been fully characterized by fractionation and methylation studies (*32*). The persistence of this polymer in kraft and semichemical pulps suggests that it is mostly part of the cellular carbohydrates, although some may be extracted directly from the wood.

Table III shows that western red alder and madrona yield a relatively pure 4-*O*-methylglucurono-xylan when extracted with 10% KOH. Numerous examples of the isolation of this type of polymer may be found in the literature; for example, alder (*Alnus rubra*) (*18*), gumwood mixture (see Table I) (*18*), elm (*Ulmus japonica*) (*50*), maple (*Acer saccharum*) (*51*), mesquite wood (*Prosposis juliflora*) (*4*), eucalyptus (*Eucalyptus regnans*) (*52*), locustwood (*Robinia pseudoacacia*) (*36*), lemonwood (*Citrus limonia*) (*36*), white birch (*Betula populifolia*) (*53*), birch (*Betula verrucosa*) (*54*), English oak (*3*), and beech (*Fagus grandifolia*) (*55*). Certain exceptions occur, however, as Jones, Merler, and Wise (*16*) found a 4-*O*-methylglucurono-araboxylan in poplar (*Populus tremuloides*), while Milks and Purves (*56*) found a 4-*O*-methylglucurono-xylan in the same wood. The structure of this latter polymer, which is found in most hardwoods, is a chain of anhydroxylose units linked by β 1 → 4 glycosidic bonds and to which are attached branches of 4-*O*-methyl-D-glucuronic acid by α 1 → 2 bonds.

For the most part, coniferous woods have been found to contain 4-*O*-methylglucurono-araboxylans in which the ratio of arabinose to xylose is about 1 to 12, and the ratio of uronic acid to xylose is about 1 to 5 or 8 (against a ratio of 1 to 8 or 12 for hardwoods in the latter case). Among the woods which have been shown to contain this polymer are western hemlock (*Tsuga heterophylla*) (*57*), European larch (*Larix decidua*) (*58*), and southern pine (an equal mixture of *Pinus elliottii* var. *elliottii* and *Pinus palustris*) (*59*). Indirect evidence indicates this same compound is present in redwood (*Sequoia sempervirens*) (*18*). Coniferous woods have also been shown to contain 4-*O*-methylglucurono-xylans. In the case of Norway spruce (*Picea excelsa*) (*60*) and loblolly pine (*Pinus taeda*) (*61*), it is not known whether they also contain the 4-*O*-methylglucurono-araboxylan as well as the 4-*O*-methylglucurono-xylan. A careful examination of earlier data (*15*) concerning the extraction of western hemlock with various alkalies indicates that a 4-*O*-methylglucurono-xylan, in addition to a 4-*O*-methylglucurono-araboxylan, is present in that wood and may be extracted with higher strength sodium hydroxide.

The alder pulp extracted here was found to contain highly soluble fractions which gave arabinose upon hydrolysis and which may be extracted from the holocellulose by the use of 10% NaOH only. Although no proof is offered, it is tempting to ascribe this result to the presence of a 4-*O*-methylglucurono-araboxylan in hardwood. Yllner and Enstrom (*62*) have similarly detected what they thought to be small amounts of a 4-*O*-methylglucurono-araboxylan, in addition to larger amounts of 4-*O*-methylglucurono-xylan, in a birchwood mixture of *Betula verrucosa* and *Betula pubescens*. These results suggest that some and perhaps all woods contain a mixture of 4-*O*-methylglucurono-xylans and 4-*O*-methylglucurono-araboxylans and that the former may predominate in hardwoods and the latter in softwoods.

The remaining resistant hemicelluloses of hardwoods have not been studied to any great extent because of their relative scarcity in the wood. A crude glucomannan fraction was isolated by Merler, Jones, and Wise (*16*) from poplar wood (*Populus tremuloides*). Methylation suggested a relatively straight chain polymer with mannose terminal units and a glucose-to-mannose ratio of about 1:2. Both western red alder and madrona holocelluloses contain fractions, resistant to extraction, which are rich in glucose and mannose. Fractionation of such a mixture from an alder semichemical pulp gave rise to six fractions, two of which were rich in glucose and mannose, two in 4-*O*-methylglucurono-xylans and two highly soluble fractions containing galactose and arabinose in addition to other sugars.

The mannose-rich fraction D was found to contain a glucomannan which had a rotation of −32° and a glucose-to-mannose ratio of 2:3. Fraction C and fraction F (see Table V) contain a polymer incorporating anhydrogalactose units. Fractionation of fraction C indicated that this polymer was not associated with mannose and might be associated with glucose. No indication is available at present to show whether this polymer is a galactan, a glucogalactan, or possibly some polymer incorporating xylose.

The most plentiful hemicelluloses of softwoods are the mannose-containing polymers. The branched galac-

toglucomannan was mentioned earlier and may be found in hemlock and southern pine holocellulose or in the conventional kraft or neutral sulphite semichemical pulps of these woods as well as in the kraft and soda pulps of redwood (*18*). The galactoglucomannan may be separated from the araboxylan of the kraft pulps (isolated by extraction with 5% NaOH) by the solubility of the galactoglucomannan triacetate in acetone or by forming its copper complex. The galactoglucomannan of the holocellulose or neutral sulphite semichemical pulp cannot be separated from the 4-*O*-methylglucurono-araboxylan of those pulps by the above processes, but may be fractionally precipitated in a pure form from 10% NaOH by the gradual addition of saturated aqueous barium hydroxide (*32*). Data published earlier (*15*) indicate that almost 80% of the mannose of western hemlock is associated with the predominantly straight chain glucomannan. This polymer, isolated from a sulphite pulp, has an optical rotation of $-37°$ and a glucose-to-mannose ratio of 1:3 (*63*). Timell and Tyminski (*64*) have isolated a similar glucomannan in high yield from white spruce (*Picea glauca*) (1:3 ratio and $-31°$). Lindberg and Meier (*65*) and Lindberg and Croon (*66*) have isolated and characterized a glucomannan from Norwegian spruce holocellulose (*Picea abies*), while Rånby and Immergut (*67*) have isolated a similar glucomannan from Norwegian spruce sulphite pulp. Glucomannans have also been isolated from western red cedar (*Thuja plicata*) (*68*), southern pine (*32*), and Sitka spruce (*Picea sitchensis*) (*69*).

The major carbohydrate constituent of hardwoods and softwoods is cellulose. E. Schulze (*70, 71*) was the first to clearly differentiate between cellulose and hemicelluloses. Since that time, numerous workers have greatly expanded the knowledge of these substances (*72*). The extraction experiments carried out here indicate that both hardwoods and softwoods can yield appreciable amounts of cellulose relatively free of extraneous polymers. These results indicate that the residual, extraction resistant sugars which remain associated with an alpha mat are not a chemical part of the cellulose but are part of inaccessible or highly insoluble polymers intimately mixed with it. Timell (*1, 2*) has compared the celluloses of hardwoods, softwoods, and grasses, primarily by viscometric techniques. These results indicate there is little difference between the celluloses from various sources other than degrees of polymerization.

The degree of polymerization of the hemicelluloses of hardwoods and softwoods has not been so extensively studied as that of cellulose. Much of the work of earlier workers is complicated by the state of purity of the samples and by the unknown degree of degradation introduced during isolation. The first work in this direction was carried out by Husemann (*73*). Glaudemans and Timell (*74*) have given an excellent review of earlier work and have also described the molecular properties of the 4-*O*-methylglucurono-xylan of white birch. It is interesting to note that Yundt (*78*) was able to crystallize low D.P. "xylans" from barley and birch, and it is possible that the original polymers may exist, in part, in a crystalline form in the original holocellulose.

A comparison of the hemicelluloses of coniferous and deciduous woods is given in Table VIII. The presence of polymers in wood other than those mentioned in the table or the text is not excluded. The presence of polymers containing xylose and 4-*O*-methyl-D-glucuronic acid joined by 1→3 glycosidic bonds has been shown by Brasch and Wise (*79, 80*). Whether these units are part of the 4-*O*-methylglucurono-xylans mentioned in this paper or part of another xylose-containing system is not known at present. The relative quantities of the hemicelluloses may be entirely different than that indicated as in the case of the arabogalactan of larch. Certainly the quantity of each hemicellulose varies from species to species and may also, in a given species, be influenced by physical environment. The composition of the wood will vary according to whether the samples are taken from sapwood, heartwood, root, branch, and even foliage. For example, a qualitative examination of needle holocellulose indicates that the carbohydrate components are different than those of the corresponding wood holocellulose (*32*). As the structures, physical properties and amounts of the various carbohydrates of wood become known, it may become more feasible to determine the actual location of the polymers in the wood fiber and perhaps to understand the function they perform in the living plant.

Table VIII. A Qualitative Comparison of the Noncellulosic Carbohydrates of Coniferous and Deciduous Woods[a]

Polymer	*Deciduous*	*Coniferous*
4-*O*-methylglucurono-xylan	Very large	Small
4-*O*-methylglucurono-araboxylan	Trace	Medium
Glucomannan	Very small	Large
Galactoglucomannan	Trace, if present	Very small
Arabogalactans	Trace, if present	Trace to medium
Other galactose-containing polymers	Trace	Trace, if present
Pectin and associated materials	Very small[b]	Very small[c]
Starch	Trace	Trace

[a] *Approximate* amouts based on percentage of the total noncellulosic carbohydrates present in wood. Very large, 80–90%; large, 60–70%; medium, 15–30%; small, 5–15%; very small, 1–5%; trace, 0.1–1%. To convert to a wood basis, an "average" figure for hemicelluloses in hardwoods would be about 25%, and a corresponding figure for softwoods would be 20%.

[b] The carbohydrate material associated with the pectin of the middle lamella upon hydrolysis gives rise to galactose, glucose, mannose, xylose, arabinose, rhamnose, and uronic acids (possibly glucuronic acid).

[c] Preliminary examination of the highly soluble carbohydrates of western red cedar indicates the probable presence of anomalous mannose-containing polymers (*68*).

CONCLUSION

This paper, in addition to comparing and contrasting the hemicelluloses of hardwoods and softwoods, has shown the existence in hardwoods of hitherto largely unexplored pectic material and accompanying polysaccharides which probably occur in the middle lamella and which are composed of galactose, glucose, mannose, arabinose, xylose, and rhamnose. The isolation of this fraction was greatly facilitated by the use of a novel chlorine dioxide-sodium bicarbonate delignification process. The main xylose-containing polymer present in hardwoods, in general, and in western red alder, in particular, was found to be a 4-*O*-methylglucuronoxylan, the over-all structure of which was characterized by the isolation of crystalline *O*-α-4-*O*-methyl-D-glucuronopyranosyl (1→2)-*O*-β-D-xylopyranosyl-(1→4)-D-xylopyranose trihydrate.

The extraction-resistant (soluble in 10% NaOH), mannose-containing polymers in hardwoods (e.g., western red alder) were shown to be glucomannans which

were different from the glucomannans of softwoods. The probable presence of a 4-*O*-methylglucurono-araboxylan in this highly inaccessible portion of the hardwood fiber was inferred as was the presence of galactose-containing hemicelluloses (i.e., perhaps a galactan or galactoglucan).

By suitable pretreatment and extraction with high-strength sodium hydroxide, chlorite holocelluloses, prepared from either type of wood, gave rise to a residue (28 to 32% yield) in which the total hemicellulosic material has been reduced to less than 0.25% for softwoods and less than 0.1% for hardwoods (based on wood). These results indicate that the residual extraction-resistant sugars which remain associated with the cellulose, as shown by hydrolysis, are not part of the cellulose but are due to inaccessible or highly insoluble polymers intimately associated with it.

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Contribution No. 44 from the Olympic Research Div., Rayonier Inc., Shelton, Wash.

The Kinetics of Oxidation of Kraft Weak Black Liquor

F. E. MURRAY

The kinetics of black-liquor oxidation have been studied over a temperature range of 122 to 196°F. and sodium sulphide concentrations up to 16 g.p.l. Oxidation rates were observed in a dynamic system using either air or pure oxygen as the gas feed to the reaction vessel. Rates of oxidation were found to vary in a complex manner depending upon the partial pressure of oxygen, the sodium sulphide concentration, the rate of liquor stirring, and the chemical reaction taking place under the prevailing experimental conditions. Results show that the rate of oxidation of weak black liquor at sodium sulphide concentrations above 2.5 g.p.l. is a maximum at a temperature between 142 and 160°F. The maximum rate in the same sulphide concentration range occurs at about 160°F. when pure oxygen is used as the gas feed to the reaction vessel. Although maximum rates occur at the indicated temperatures, the change in rate with temperature is actually very slight. Under some conditions, elemental sulphur is a product of the oxidation reaction. Its formation is favored by low temperature, low sulphide concentration, and high oxygen pressures. The sulphur formed goes into solution during storage to regenerate some sulphide. This reversion of oxidized sulphide can, however, be avoided by proper choice of operating conditions. Equations were developed to describe the oxidation rate in terms of sulphide concentration and oxygen pressure. Data on rates obtained in the laboratory apparatus are compared with results obtained from studies of pilot plant and full-scale oxidation units.

BLACK-liquor oxidation is accepted generally as the most promising means of curtailing malodor from kraft pulping operations. Although the benefits of the process have been known for many years (*1*), the only published data on reaction kinetics (*2*) deal with a special case involving very large liquor surface and high oxygen pressure. Results described in this paper indicate that experimental conditions in the previous work (*2*) represent a limiting case, in which the chemical reaction rate governs the over-all rate of sulphide oxidation. This work was begun to obtain general knowledge on all phases of black-liquor oxidation. This first stage of the program involves experiments to study the influences of sulphide concentration, temperature, oxygen pressure, and liquor turbulence on the oxidation rate.

EXPERIMENTAL PROCEDURES

Samples

The weak black liquor was obtained from a west coast kraft mill. It contained about 9.5 g.p.l. of Na_2S before oxidation. In the sampling procedure, 45 gal. of black liquor were run into a cleaned steel drum. The drum was sealed and the liquor allowed to cool. The cool liquor was then used to fill 28 one-gal. glass jugs and these were tightly sealed to exclude air. During several weeks of storage, portions of this 28-gal. sample gave reproducible results under identical experimental conditions. However, exposure to air for extended periods at room temperature resulted in decreased oxidation rates in the liquor.

F. E. MURRAY, British Columbia Research Council, Vancouver, B. C.

Apparatus

The apparatus used in the work is shown in Fig. 1 and 2. The experimental oxidations were carried out in a stainless steel reaction vessel (8) about 1 sq. ft. in cross section. This vessel was 80% immersed in an oil-bath thermostat.

For an experimental run, 4 l. of black liquor, containing about 9.5 g.p.l. of Na_2S were heated to the desired reaction temperature in a covered stainless steel beaker. The hot liquor was then added to the reaction vessel to give a liquor depth of about 2 in. During an experiment, the liquor was stirred at a constant rate by rotation of the perforated stainless steel disk (9) with the variable speed motor (4). The tachometer (5) was used to measure the rate of rotation of the liquor stirrer. Unless otherwise noted in the text, all experiments were done with this disk rotating at 41.7 r.p.m.

The gas phase in the reaction vessel was vigorously agitated by the gas stirrer (10) which was driven through the pulley (12) by the high speed motor (6).

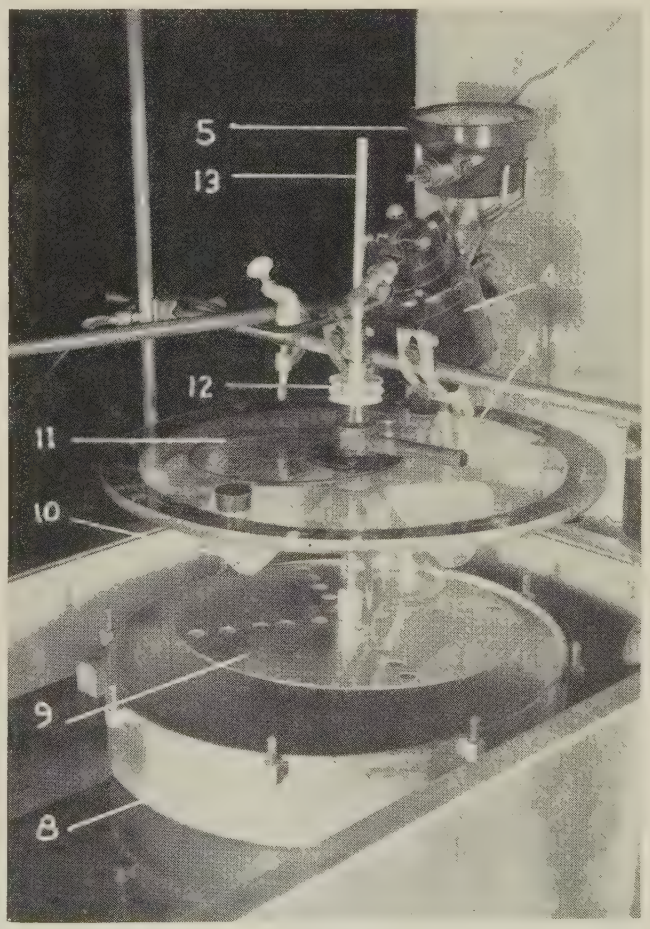

Fig. 1. The reaction vessel for observing oxidation rates

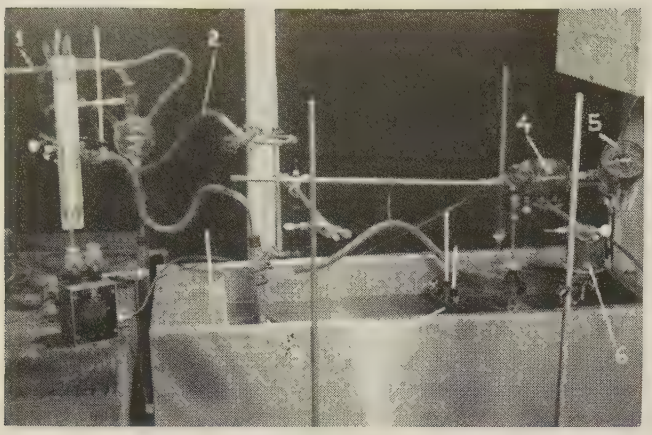

Fig. 2. Arrangement of the components in the apparatus

Nylon bushings were used throughout to avoid contamination of the liquor by lubricants.

Air from a compressor or pure oxygen from a cylinder was metered through the orifice meter (1) and passed into the glass mixing bulb. In the mixing bulb, the air or oxygen was saturated with water vapor and heated using steam from line (2). Flow of air or oxygen was maintained constant and the temperature of the gas-steam mixture leaving the mixing bulb was kept at the temperature of the reaction vessel by controlling the steam flow. The mixture of gas and water vapor next passed through the large flask (3) immersed in the thermostat. This flask removed entrained mist and aided in equalizing the gas feed temperature with that of the thermostat and reaction vessel. From the flask (3), the gas feed passed into the reaction vessel through the central inlet tube (14). Gas and vapor from the reaction vessel were discharged through an opening near the outer edge of the Lucite cover (11).

The air or oxygen flow rates to the reaction vessel were chosen sufficiently high to maintain a virtually constant oxygen pressure throughout any experimental run. Liquor samples were withdrawn at 15-min. intervals and analyzed for sodium sulphide concentration using a colorimetric method (*3*).

RESULTS

Oxidation Rates

The rate of oxidation of sodium sulphide in black liquor was found to vary with temperature, with sulphide concentration, with oxygen partial pressure and with the liquor stirring rate.

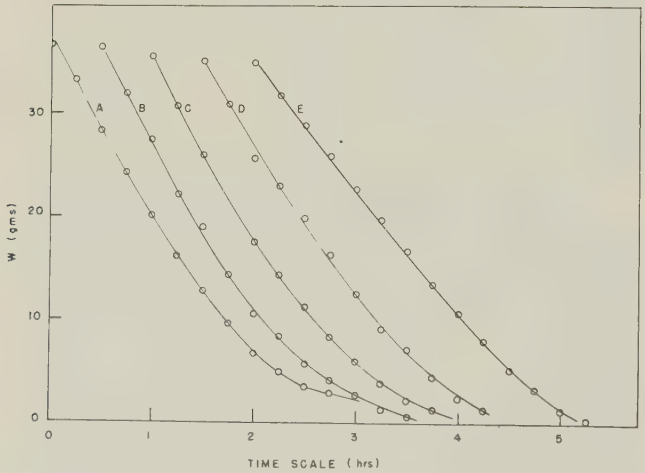

Fig. 3. The weight of sodium sulphide in solution as a function of time when oxidizing with air feed. Curve A at 122°F., B at 141°F., C at 160°F., D at 175°F., and E at 196°F.

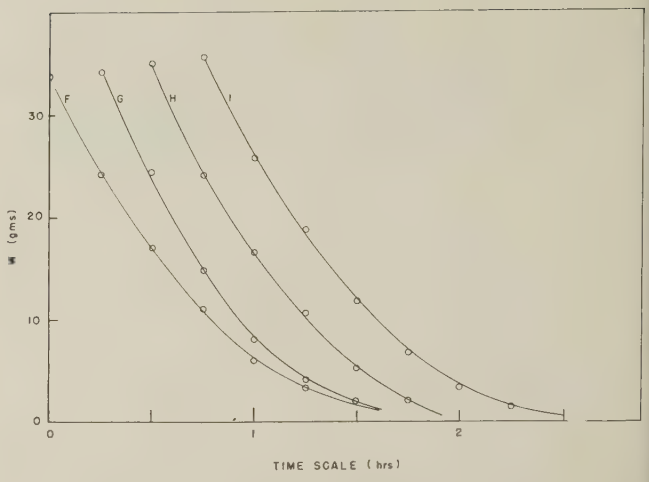

Fig. 4. The weight of sodium sulphide in solution as a function of time when oxidizing with a pure oxygen feed. Curve F at 141°F., G at 160°F., H at 175°F., and E at 196°F.

The total weights of sodium sulphide remaining in solution at various times during oxidation runs are shown in Figs. 3, 4, and 5. The origin of the time scale for each experiment is displaced horizontally for easier comparison of slopes and curvatures. The slope of a curve at any point gives the oxidation rate and the curvature yields information on the reaction mechanism at the prevailing temperature and oxygen pressure. Values of the oxidation rates for sodium sulphide concentrations of 2.5, 5, and 7.5 g.p.l. are given in Table I.

In Fig. 3, curves A, B, C, D, and E show results for oxidation using air at temperatures of 122, 141, 160, 175, and 196°F., respectively. Examination of these curves along with the data in Table I shows that the oxidation rate changes only slightly with temperature when the sodium sulphide concentration is greater than 1 g.p.l. Below this concentration, the rates of oxidation decrease rapidly with decrease of temperature. At the higher sulphide concentration, the oxidation rate passes through a maximum between 141 and 160°F.

The curvature of the lines in Fig. 3 shows a marked variation with temperature indicating that changes in the mechanism of oxidation are occurring. Thus,

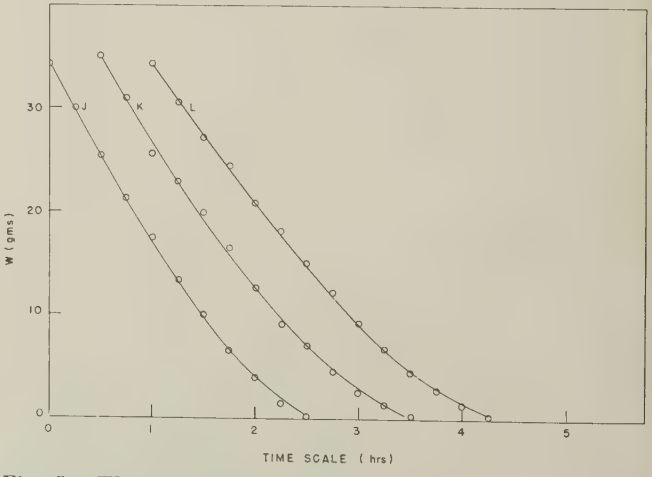

Fig. 5. The weight of sodium sulphide in solution as a function of time with air feed at 175°F. and different liquor stirring rates. Curve J at 83.4 r.p.m., K at 41.7 r.p.m., and L at 21.0 r.p.m.

curve E, at 196°F., is almost a straight line throughout showing that the oxidation rate, using air at this temperature, is only slightly affected by changes in sulphide concentration. Curves B and C, however, show that

Table I. Values of *dW/dt* at Various Temperatures and Sodium Sulphide Concentrations

Gas feed C, *g./l.*	*Air* 2.5	5.0	7.5	*Oxygen* 2.5	5.0	7.5
Temp., °F.						
122	12.1	15.0	17.5	...	...	...
141	11.3	16.0	19.3	19.9	30.1	37.7
160	11.0	15.7	18.8	24.2	37.8	45.3
175	11.4	14.8	16.0	21.2	31.4	44.0
196	11.4	11.9	12.1	19.5	30.1	37.7

oxidation rates at 141 and 160°F. increase appreciably with increasing sulphide concentration. These observations indicate that oxygen pressure (which remains constant throughout any experiment) is the dominant factor in limiting the oxidation rate using air at 196°F. At lower temperatures (higher oxygen pressures) the oxidation rate is more dependent on sulphide concentration.

Table II. Ratio of Oxidation Rates with Oxygen to Those Obtained Using Air as Gas Feed

C, *g./l.* →	2.5	5.0	7.5
Temp., °F.			
141	1.76	1.88	1.95
160	2.20	2.41	2.41
175	1.86	2.12	2.75
196	1.71	2.53	3.11

A comparison of curves E and I also shows that the influence of sulphide concentration on the oxidation rate increases as the oxygen pressure is increased. Both experiments were conducted at 196°F. but the oxygen pressure was changed by a factor of five. When pure oxygen was fed to the apparatus (curve I), the oxidation rate changed appreciably with changing sulphide concentration. With air feed (curve E) the rate of oxidation remained almost constant over the entire range of sulphide concentrations.

Table III. Values of *dW/dt* at Three Liquor Stirring Rates at 175°F. Using Air Feed

C, *g./l.* →	2.5	5.0	7.5
Stirring rate, r.p.m.			
21	10.8	12.3	13.1
41.7	11.4	14.8	16 0
83.4	13.1	16.0	18.8

Curves F, G, H, and I plotted in Fig. 4 were determined using pure oxygen at 141, 160, 175, and 196°F., respectively. At sodium sulphide concentrations above 2 g.p.l. the oxidation rate was a maximum at 160°F. Oxidation rates using oxygen were about double those obtained using air at the same temperature (Table I). Thus, a fivefold increase in oxygen pressure does not cause a corresponding increase in oxidation rate. The ratios of oxidation rates using pure oxygen to those obtained using air under similar conditions are shown, at a number of temperatures and sulphide concentrations, in Table II. Although the ratios show considerable scatter, they tend to increase as oxygen pressure becomes more dominant in controlling the oxidation rate. Consequently, the ratio tends to increase with increasing temperature at a given sulphide concentration and increase with increasing sulphide concentration at a fixed temperature.

The effect of liquor turbulence on the oxidation rate is shown by the three curves in Fig 5. Oxidation rates obtained from these curves are given in Table III. Curves J, K, and L were obtained at 175°F. and stirring rates of 83.4, 41.7, and 21.0 r.p.m., respectively. The oxidation rate was increased substantially by increasing the turbulence in the black liquor. This observation was expected because the absorption of oxygen is generally a liquid-film-controlled process.

Table IV. Regeneration of Sulphide in Oxidized Black Liquor Stored for 12 Hr. at 185°F.

Oxidation temp., °F.	*Gas feed*	*Initial C in oxidized liquor*	*C in oxidized liquor after storing*	*Total Na_2S reverted,*[a] *%*
122	Air	0.90	2.46	18.2
141	Air	0.27	1.10	9.0
160	Air	0.18	0.27	1.0
175	Air	0.04	0.29	2.5
196	Air	0.01	0.08	0.8
141	O_2	0.25	1.06	9.0
160	O_2	0.33	0.72	4.5
175	O_2	0.00	0.06	0.7
196	O_2	0.02	0.12	1.0

[a] Based on Na_2S concentration before oxidation of 9.5 g. per liter.

Regeneration of Sulphide after Oxidation

Under certain experimental conditions, one of the oxidation products was elemental sulphur which appeared as a visible film on the surface of the black liquor. Its formation was favored by low temperatures, high oxygen pressures, and low sulphide concentrations.

With air as the gas feed, no formation of sulphur was observed in any experiment at or above 160°F. At 141°F., a visible amount of sulphur began to accumulate when the sodium sulphide concentration had fallen to 1.5 g.p.l. At 122°F., sulphur began to form when the sodium sulphide concentration had fallen to 3.5 g.p.l.

With oxygen as the gas feed, no formation of sulphur was observed in any run at or above 175°F. At 160°F., visible amounts of sulphur began to accumulate when the sodium sulphide concentration in the liquor had fallen to 2 g.p.l. At 142°F., sulphur formed throughout the experiment at all concentrations.

Since sulphur in alkaline solution is unstable with respect to the formation of both sulphide and thiosulphate, it was expected that part of the sulphur would revert to sulphide in some of the samples during storage of the oxidized liquor. Samples of the oxidized liquor were stored under methane and heated in the absence of air for 12 hr. at 185°F. Each sample was analyzed for sulphide at the end of the 12-hr. period. The percentage of the oxidized sulphide which reverted was calculated on the basis of an initial concentration of 9.5 g.p.l. of Na_2S in the unoxidized black liquor. Results are shown in Table IV.

Miscellaneous Observations

Several experiments were conducted to obtain general information about the oxidation process. In one experiment, the oxidation of weak black liquor with air feed at 160°F. was allowed to proceed normally to a sodium sulphide concentration of about 5 g.p.l. Then, 100 g. of solid $Na_2S_2O_3$ were added to the black liquor. Addition of the thiosulphate was followed by a definite decrease in the oxidation rate.

In a similar experiment, 100 g. of NaOH were added to

another portion of partially oxidized black liquor. Addition of the sodium hydroxide was followed by an increase in the oxidation rate.

Several experiments using more concentrated liquor containing 30% solids showed that the oxidation rates are not greatly affected by change in solids content. In a number of experiments, the sodium sulphide concentration in the weak black liquor was increased to about 20 g.p.l. by addition of technical grade sodium sulphide. Oxidation of sulphide in the resulting solution proceeded at a lower rate for a given sulphide concentration than in unmodified black liquor. This effect may be caused by the presence of elemental sulphur or thiosulphate in the added sodium sulphide, or by the change in the caustic-sulphide ratio when the sodium sulphide is added to the weak black liquor.

DISCUSSION

Mechanism of Oxidation

The oxidation of weak black liquor with air cannot be described by any simple chemical or absorption rate theory. The complexity of the oxidation results because the temperature region of practical interest is a transition zone in which the products of the chemical reaction, the sulphide concentration and the oxygen partial pressure act together to influence the oxidation rate.

However, the reaction mechanism can be more clearly understood and useful practical equations obtained starting with the semiempirical relationship:

$$\frac{dW}{dt} = K(C + bP) \qquad (1)$$

(All symbols used are defined in section E.) Equation (1) indicates that the oxidation rate depends partly on C, the sodium sulphide concentration in the liquor which affects the rate of the chemical reaction, and partly upon the rate of oxygen absorption which, in turn, is dependent upon its partial pressure P.

During any experimental run, the volume of black liquor in the reaction vessel changed slightly as samples were taken for analysis and as a result of small imbalances in the evaporation-condensation rates at the black liquor surface. This volume change reached a maximum of about 3% during some extended runs.

If the volume change during an experiment is small compared to the changes in the reaction rate, equation (1) may be integrated to the form:

$$V \log (C + bP) = \frac{K}{2.30} b + B \qquad (2)$$

If the volume change in an experiment is appreciable compared to the changes in the reaction rate, equation (1) must be integrated to the more exact form:

$$\log (C + bP) = \frac{K}{\alpha} \log V + D \qquad (3)$$

Derivation of equation (3) is given in the Appendix.

In the oxidation of black liquor, the parameters K and b are constants only for given temperature, stirring rate of the liquor and oxygen pressure in the reaction vessel. However, the value of b reflects the importance of the oxygen absorption rate in determining the over-all oxidation rate. If b is large, the oxygen absorption rate limits the over-all oxidation rate; if b is small, the chemical reaction rate becomes an important factor in controlling the over-all oxidation rate.

Table V. Variation of K and b with Temperature, Oxygen Pressure and Liquor Stirring Rates

Liquor stirring rate, r.p.m.	*Gas feed*	*Temp., °F.*	P	K	b	bP
41.7	Air	122	0.175	1.14	48.4	8.45
41.7	Air	141	0.160	1.60	29.5	4.72
41.7	Air	160	0.136	1.56	34.5	4.70
41.7	Air	175	0.108	0.98	85.0	9.18
41.7	Air	196	0.056	0.14	1417	79.40
41.7	O_2	141	0.799	3.66	4.05	3.24
41.7	O_2	160	0.680	4.16	5.10	3.47
41.7	O_2	175	0.540	4.65	3.81	2.06
41.7	O_2	196	0.281	3.70	10.7	3.00
21.0	Air	175	0.108	0.47	197	21.30
41.7	Air	175	0.108	0.98	85.0	9.18
83.4	Air	175	0.108	1.14	83.3	9.00

Values of K and b at various temperatures and oxygen pressures are given in Table V. With oxygen feed to the reaction vessel, b is relatively small and increases appreciably only at 196°F. This indicates that sulphide concentration is an important rate-controlling factor when oxygen is used. Using air as the gas feed to the apparatus, values of b are much larger and increase rapidly with increasing temperature. At the low oxygen pressures prevailing with air feed, the over-all oxidation rate shows a strong dependence on oxygen pressure.

Values of K and b were obtained by fitting equation (2) or (3) to the observed rate data. Details of this method are given in the Appendix. Typical graphs of data calculated using equations (2) or (3) are shown in Figs. 6, 7, and 8. Values of K were calculated from the slopes of the straight lines. Fig. 6 shows results calcu-

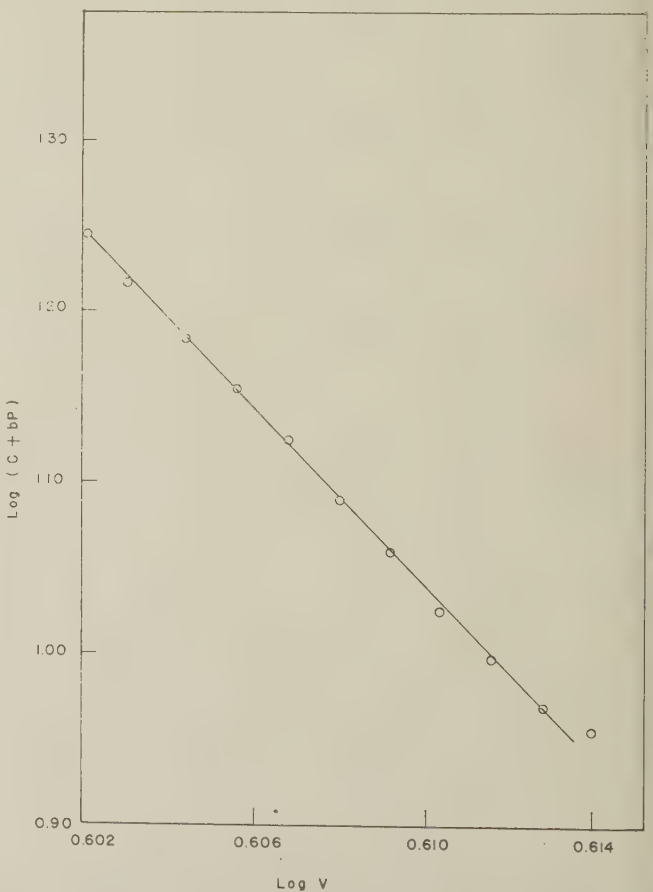

Fig. 6. A plot of log (C + bP) vs. log V at 175°F. using air feed and liquor stirring rate of 83.4 r.p.m.

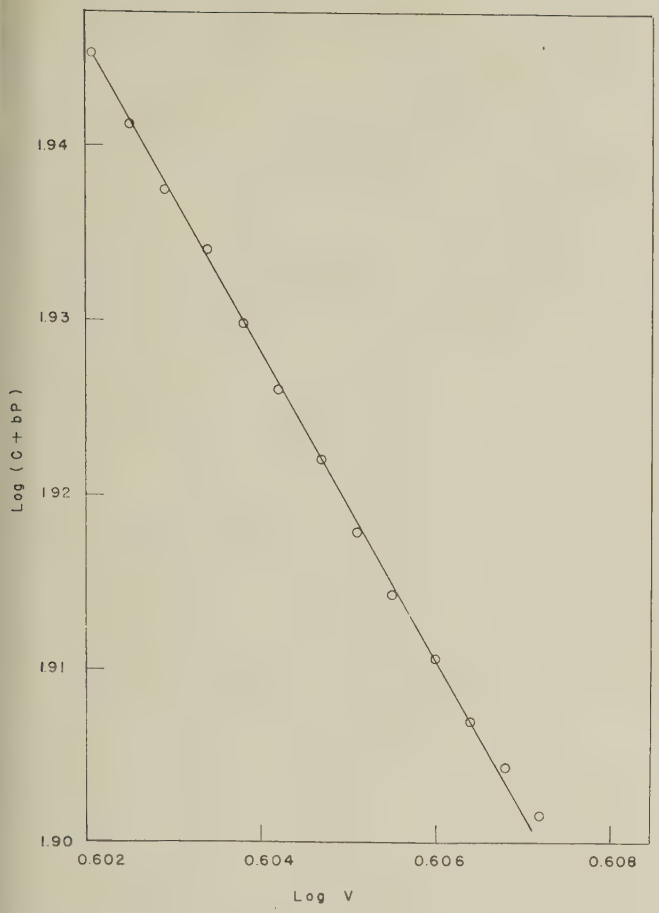

Fig. 7. A plot of log (C + bP) *against log V at 196°F. using air feed and liquor stirring rate of 41.7 r.p.m.*

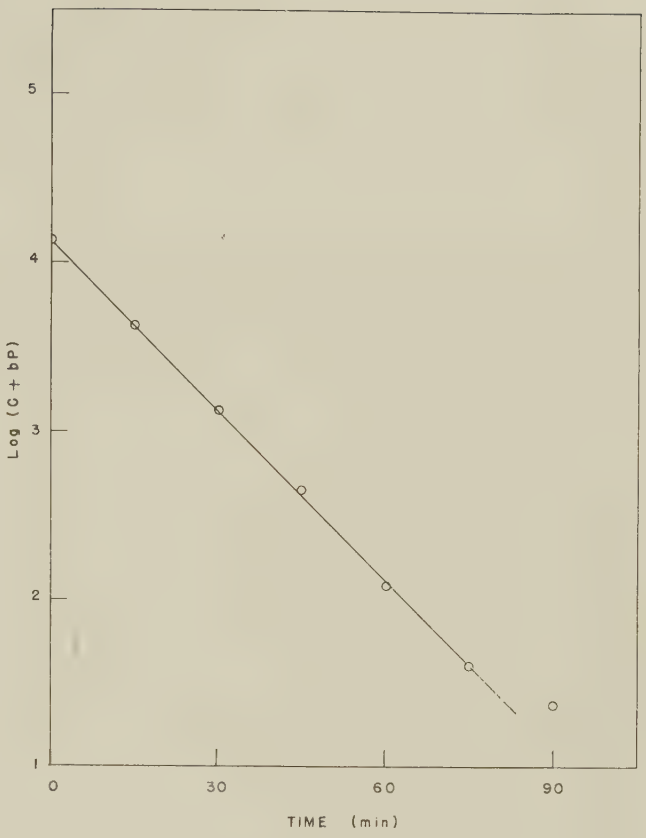

Fig. 8. A plot of log (C + bP) *against time at 175°F. using pure oxygen feed and liquor stirring rate of 41.7 r.p.m.*

lated from data obtained at 175°F. using air feed and a stirring rate of 83.4 r.p.m. Fig. 7 refers to data obtained using air feed at 196°F. Results plotted in Fig. 8 were obtained using a pure oxygen feed at 175°F.

With air feed to the apparatus, values of K and b were constants above a sodium sulphide concentration of 0.5 g.p.l. except in cases where sulphur was a product of oxidation. At sodium sulphide concentrations below 0.5 g.p.l., oxidation rates observed were somewhat lower than those calculated using the best-fit equation. Thus the points near the horizontal axis in Figs. 6, 7, and 8 are above the straight line which fits the rest of the points. In the oxidation of black liquor with air, in the temperature region of practical interest, equation (1) can be used with appropriate values of K and b to give the oxidation rate over a concentration range from 9.5 to less than 0.5 g.p.l.

Values of $\frac{dW}{dt}$ were obtained directly from the curves in Figs. 3, 4, and 5 at sodium sulphide concentrations of 2.5, 5.0, and 7.5 g.p.l. In Table VI, values of $\frac{dW}{dt}$ from Fig. 3 are compared to those calculated using equation (1) with values of K and b obtained as described in the Appendix.

Table VI. Comparison of Calculated Values of *dW/dt* with Those Obtained Directly from the Curves in Fig. 3

	Values of dW/dt *obtained directly at three concentration levels*			*Values of* dW/dt *calculated from equation (1) at three concentration levels*		
C, *g./l.* →	*2.5*	*5.0*	*7.5*	*2.5*	*5.0*	*7.5*
Temp., °F.						
122	12.1	15.0	17.5	12.5	15.3	18.3
141	11.3	16.0	19.3	11.6	15.6	19.5
160	11.0	15.7	18.8	11.2	15.1	19.0
175	11.4	14.8	16.0	11.4	14.5	17.0
196	11.4	11.9	12.1	11.5	11.8	12.2

Practical Significance of Results

1. *Conditions for Most Effective Oxidation Rates.* At sodium sulphide concentrations above 2.5 g.p.l., the maximum oxidation rate (without sulphur formation) was observed at 160°F. At lower sulphide concentrations, oxidation was most rapid at 196°F.

These facts indicate that a two-stage oxidation would be most effective in oxidizing the sulphide in black liquor. The first stage would involve partial oxidation of weak black liquor at temperatures of about 160 to 175°F. The second stage could be carried out using partially concentrated liquor at a higher temperature. In this way the oxidation process could achieve its optimum effectiveness as an odor control and chemical recovery operation.

2. *Reversion of Oxidized Sulphide.* Reversion of oxidized sulphide may occur to an appreciable extent if one of the products of oxidation is elemental sulphur. In a kraft mill, reversion in weak black liquor would occur in the storage tanks and evaporators and would partially nullify the benefits of oxidation. However, this reversion may be avoided by operating oxidation equipment at temperatures of 160°F. or higher.

3. *Comparison of Laboratory Results with Operating Data.* It is of interest to compare values of K and $\frac{dW}{dt}$ obtained from data on operating units and pilot plant equipment with those obtained in these laboratory studies.

The average temperature in a tower oxidizing weak black liquor is about 175°F. At this temperature and the highest stirring rate used in the laboratory work, $b = 83.3$ and $bP = 9.00$. The oxidation rate per unit area is then given by the equation

$$\frac{dW}{dt} = K(C + 9.00)$$

From the material balance for sodium sulphide in a volume element dh of a packed oxidation tower (*4*)

$$LdC = Ka(C + 9.00)dh$$

Integrating this equation over the height of the packing in the tower, one obtains the relationship:

$$\log \frac{C_1 + 9}{C_2 + 9} = \frac{Ka}{2.30L} h$$

Table VII shows comparative values of K and dW/dt for two types of asbestos-cement packing and the laboratory apparatus. Oxidation rates in packed towers were expected to be somewhat higher than laboratory values as a result of the greater liquor turbulence on the packing sheets.

Table VII. Comparison Between Values of *K*, *Ka*, and *dW/dt* Obtained in Towers Packed with Vertical Asbestos Cement Plates and Those Obtained in the Laboratory Oxidation Apparatus

Mean temperature in all cases = 175°F.

Stirring rate in reaction vessel, r.p.m.	*Type of packing*	*Value of L in packed unit*	K	Ka	dW/dt at C = 5
41.7	...	...	0.98	...	13.9
83.4	...	...	1.14	...	16.0
...	Corrugated	1630	0.97	35.0	13.6
...	Corrugated	2450	1.14	41.2	16.0
...	Corrugated	3270	1.26	45.4	17.6
...	Straight	1700	1.02	24.6	14.3
...	Straight	2040	1.14	27.3	16.0
...	Straight	2380	1.37	32.9	19.2

4. *Effect of Chemical Additions on Reaction Rate.* Experiments showed that an increase in thiosulphate concentration in the black liquor reduced the oxidation rate while an increase in sodium hydroxide concentration increased the oxidation rate.

These results indicate that liquors having low sulphidities will oxidize more rapidly for given sulphide concentration than high sulphidity liquors.

The addition of elemental sulphur to digesters produces sulphide according to the equation:

$$4S + 6NaOH \rightarrow 2Na_2S + Na_2S_2O_3 + 3H_2O$$

The over-all effect is to lower the sodium hydroxide concentration in the black liquor and increase the thiosulphate concentration. Both these changes adversely affect the oxidation rates in the black liquor for given sulphide concentration, although the decrease in rate may be slight.

NOMENCLATURE

- a = Surface area per unit packed tower volume, sq. ft. per cu. ft.
- α = rate of change of black liquor volume in the reaction vessel, l. per hr.
- b = parameter in oxidation rate equation, g.p.l. atmosphere
- B = constant of integration
- C = concentration of sodium sulphide in black liquor, g.p.l.
- C_1 = concentration of sodium sulphide in black liquor at the inlet to oxidation unit, g.p.l.
- C_2 = concentration at outlet
- D = constant of integration
- h = height of packing in an oxidation tower, ft.
- K = parameter in the oxidation rate equation, l. per hr.-sq. ft.
- Ka = capacity coefficient for a tower packing, l. per hr.-cu. ft.
- L = liquor feed density to an oxidation tower, l. per hr.-sq. ft.
- P = partial pressure of oxygen, atm.
- t = time, hr.
- W = total weight of sodium sulphide in the reaction vessel at any time, g.
- $\frac{dW}{dt}$ = oxidation rate for sodium sulphide in black liquor, g. per hr.-sq. ft.

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APPENDIX

Equation (1) in the text is written

$$\frac{dW}{dt} = K(C + bP) \qquad (1)$$

Since $W = VC$, equation (1) may be written

$$VdC + CdV = K(C + bP)dt \qquad (4)$$

During any experimental run, V changed with t according to the equation:

$$V = (4.000 + \alpha t) \qquad (5)$$

Substituting for V in equation (4),

$$(4 + \alpha t)dC = K\left[\left(1 + \frac{\alpha}{K}\right) C + bP\right] dt \qquad (6)$$

The term α/K is generally very much smaller than unity so for practical purposes, equation (6) may be written

$$(4 + \alpha t)dC = K(C + bP)dt \qquad (7)$$

Integration of equation (7) yields the result

$$\log (C + bP) = \frac{K}{\alpha} \log (4 + \alpha t) + D \qquad (8)$$

But $(4 + \alpha t) = V$ so that equation (8) becomes

$$\log (C + bP) = \frac{K}{\alpha} \log V + D \qquad (3)$$

If $\log (C + bP)$ is plotted as the ordinate against $\log V$, the slope of the straight line obtained is equal to K/α. Since α is known experimentally, K can be readily calculated.

Values of K and b for each experimental run were obtained in the following manner: From Figs. 3, 4, and 5, values of dW/dt were obtained by drawing tangents to each curve at points corresponding to sodium sulphide concentrations of 2.5, 5, and 7.5 g.p.l. Approximate values of K and b were calculated for each curve using these values of dW/dt.

The values of b obtained in this manner were substituted in equation (2) or (3). If the points obtained in the experiment fell on a straight line, using this value of b, it was accepted as correct. If the experimental points did not fall on a straight line, b was changed to

obtain a best-fit straight line by a series of graphical trials.

When the value of b used in plotting the experimental points yielded the desired best-fit straight line, K was obtained from the slope of this line. Typical graphs used to obtain K and b values are shown in Figs. 6, 7, and 8.

This procedure was much easier than it appears as values of b calculated from the tangents were either the best values or very near to them.

Secondary Motions in Flow Between Concentric Rotating Cylinders

DAVID W. APPEL

The results of a study of fully developed secondary motions in the flow between concentric cylinders, with the inner one rotating, are reported herein. These motions, called Taylor vortices, have been shown to have important effects on the flow through fourdrinier paper machines. Existence of both laminar and turbulent regimes of flow in Taylor vortices was established. The strength of the vortices was measured and found to increase linearly with increase in the rotational speed of the inner cylinder up to the point of transition to turbulent motion. Thereafter, the strength of the vortices was found to reach a maximum and decrease with further increase of the speed of the cylinder. The effects of adding an axial discharge between the cylinders were also studied. Under certain conditions, boundary layers formed on the cylinder walls due to the through-flow, thereby confining the Taylor vortices to the central part of the annulus. However, with higher cylinder speeds and correspondingly stronger vortices, these boundary layers were obliterated. From this study a better understanding of this type of secondary flow has been obtained. The results are presented dimensionlessly to facilitate their application to problems encountered in the flow through paper machines.

THE stability of flows with curvature has long been of interest to hydrodynamicists, and has recently received considerable attention in the paper industry since the importance of secondary motions in flow through fourdrinier paper machines has become known. Theoretical predictions of the onset of secondary motions have been made for a number of different boundary conditions, and these have been well substantiated experimentally. At the same time an increasing need has developed for quantitative information on the secondary motions after they are fully established. The purpose of the study described herein was to obtain information on a particularly well-defined case of secondary motion, that occurring in an annulus between a rotating inner cylinder and a stationary concentric outer cylinder. The criterion for onset of this type of flow was fully determined by Taylor in a work that is recognized as classic (*1*). However, only the gross aspects of the secondary motion, now known as Taylor vortices, were observed. In the present study, the strength of Taylor vortices, the type of flow within Taylor vortices, and the effect of a superimposed axial discharge between the cylinders were determined. The results may be applied directly in problems concerning incompressible viscous flows between rotating cylinders and, perhaps more importantly, the information may serve as a guide for both theoretical analysis and design in other more complicated flows involving a secondary motion due to normal acceleration of the flow.

DAVID W. APPEL, Associate Professor, Dept. of Engineering Mechanics, University of Kansas, Lawrence, Kan.

BACKGROUND ON TAYLOR VORTICES

The criterion for stability of a curvilinear flow with negligible viscous effects was first stated by Lord Rayleigh (*2*) that the product of velocity and radius must be an increasing quantity with increasing radius. For a fluid between a pair of coaxial cylinders with the inner one rotating, as shown in Fig. 1, the velocity distribution for all cylinder speeds gives rise to an unstable condition of flow according to this criterion. However, with viscous effects present, Taylor showed that secondary motions would not occur until a certain critical Reynolds number was reached. For the particular case in which only the inner cylinder rotates, Taylor's theory indicated that

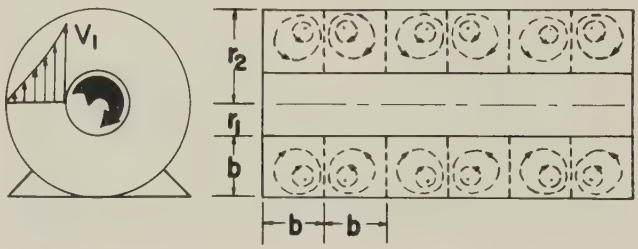


Fig. 1. Secondary flow pattern in an annulus

$$R_c = \left(\frac{V_1 b}{\nu}\right)_c = 41.2\sqrt{\frac{r_1 + r_2}{2b}}$$

in which

R_c = critical Reynolds number
V_1 = velocity of a point on the inner cylinder
ν = kinematic viscosity of the fluid
r_1 = radius of the inner cylinder
r_2 = radius of the outer cylinder
b = width of the annulus, $r_2 - r_1$

For flow at a Reynolds number greater than this critical, a secondary motion occurs having the form of a series of ring vortices filling the annulus between the cylinders, as shown in the side view of the cylinders in Fig. 1. These vortices fill compartments which are approximately square in cross section. Adjacent vortices have opposite directions of rotation so that the flow between one pair will be radially outward, and between the next pair will be radially inward. Once this new pattern of motion is established the flow is once again stable and

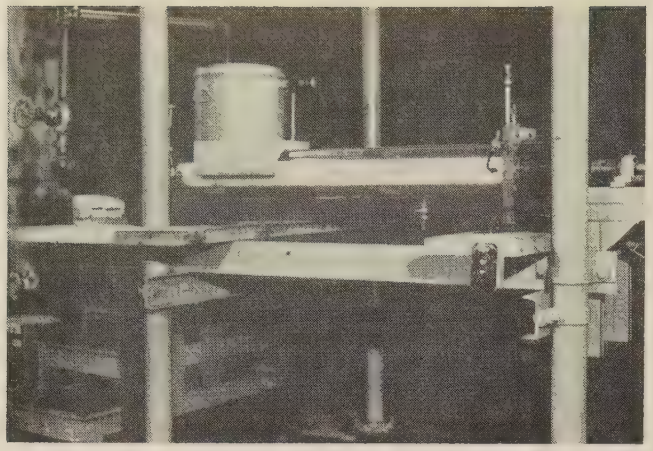


Fig. 2. Experimental equipment

the vortices remain unchanged as long as the inner cylinder rotates at a constant speed.

Taylor vortices were found to be one cause of streaks in paper made on fourdrinier machines in studies by Lee. (*3*). Also, in laboratory tests using both glycerol-water solutions and suspensions of paper pulp, Lee was able to check closely the critical Reynolds number for onset of vortex motion predicted by Taylor. Thus, results of laboratory experiments using glycerol-water mixtures are believed to be applicable to flows of pulp in the range of Reynolds numbers of the tests described herein.

In contrast to the extensive attention given to the stability of viscous flow between rotating cylinders, little information had been obtained on the secondary motions once they were established. Taylor noted simply that: "A moderate increase in the speed of the apparatus merely increased the vigor of the circulation in the vortices without altering appreciably their spacing or position, but a large increase caused the symmetrical motion to break down into some kind of turbulent motion which was impossible to follow by eye." Quantitative data on the variation in strength of the vortices with inner cylinder speed, the transition to turbulent motion in the vortices, and the effects of an axial discharge on the formation of the vortices were needed. It was these aspects of the flow between rotating cylinders that were selected for the present study.

EQUIPMENT FOR LABORATORY STUDY

The principal equipment used in this study was built in the Fluid Mechanics Laboratory of the Kimberly-Clark Corp. and is shown in Fig. 2. The outer cylinder was of clear plastic 5.5 in. i.d. and approximately 6 ft. long over-all. Three inner cylinders, 2.25, 4.12, and 5.37 in. diam. and 4 ft. long, were driven by a variable-speed transmission at speeds from 50 to 600 r.p.m. The inner cylinders were painted white to contrast with dyed fluid which was injected through a hypodermic needle into the annulus for visual and photographic observation of the flow. A similar set of shorter concentric cylinders was assembled in the Fluid Mechanics Laboratory at the University of Kansas. The inner cylinders were 1.25 and 3.00 in. diam. and, to eliminate flexing when they rotated, their length was made 18 in. The inside diameter of the outer cylinder was 5.5 in. and the length 2 ft. In other respects the cylinders and drives for the inner cylinders were essentially the same in the two sets of equipment.

Pipe connections to a pump and reservoir were made to provide an axial flow between the cylinders. Also, a water box, located on top of the cylinders at the left hand side of Fig. 2 provided an undistorted view of the flow for some special tests.

In all tests, solutions of glycerol in water were used as the fluid medium. The specific gravity of each mixture was measured and then from published data on these solutions (*4*) the percentage glycerol and the viscosity at the temperature of each test were determined. For a few tests at elevated temperatures additional data on viscosity were obtained using an Ostwald viscosimeter.

For measurements of the strength of Taylor vortices, a special spinner was made of shim brass 0.008 in. in thickness, cut and bent to form four vanes in two perpendicular planes passing through the axis of rotation as shown in Fig. 3. The over-all diameter of the spinner was $^3/_8$ in. Mechanical friction was minimized by mounting a precision jewel bearing in the hub of the spinner. A shaft intended for a second hand of a watch was passed through the jewel and cemented into the end of a bent brass rod which served as a support for the the spinner. With the spinner in a uniform axial flow no marked tendency toward rotation was observed. However, even slight blowing on one vane caused the spinner to rotate freely. Mechanical friction was very small and was assumed to be negligible.

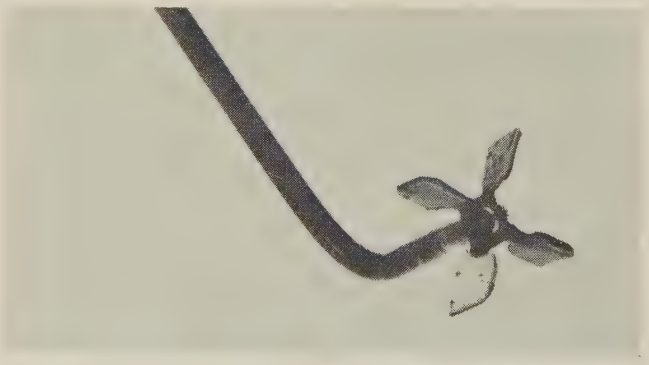


Fig. 3. Spinner for measurement of vortex strength

MOTION WITHIN TAYLOR VORTICES

One important feature of established Taylor vortices apparently not noticed by earlier observers is their

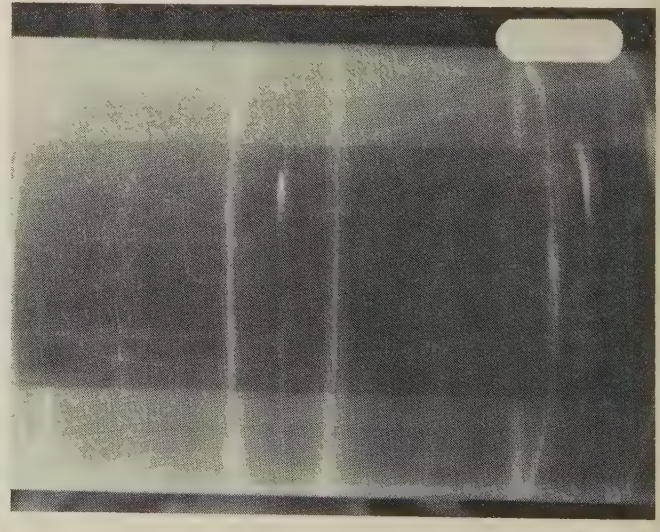


Fig. 4. Air rings in Taylor vortices

marked asymmetry. This was most easily seen when a small amount of air was introduced into the flow and allowed to migrate to the region of minimum pressure

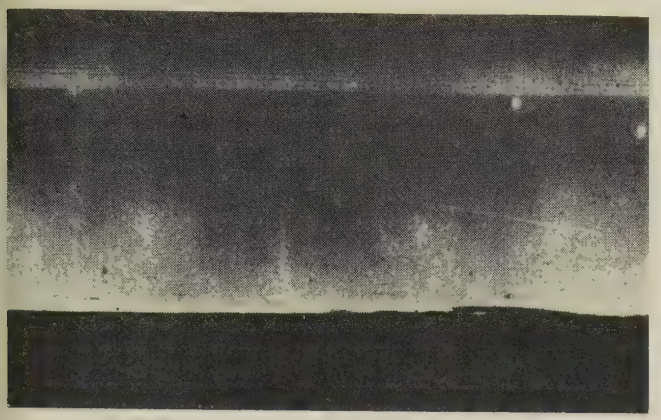
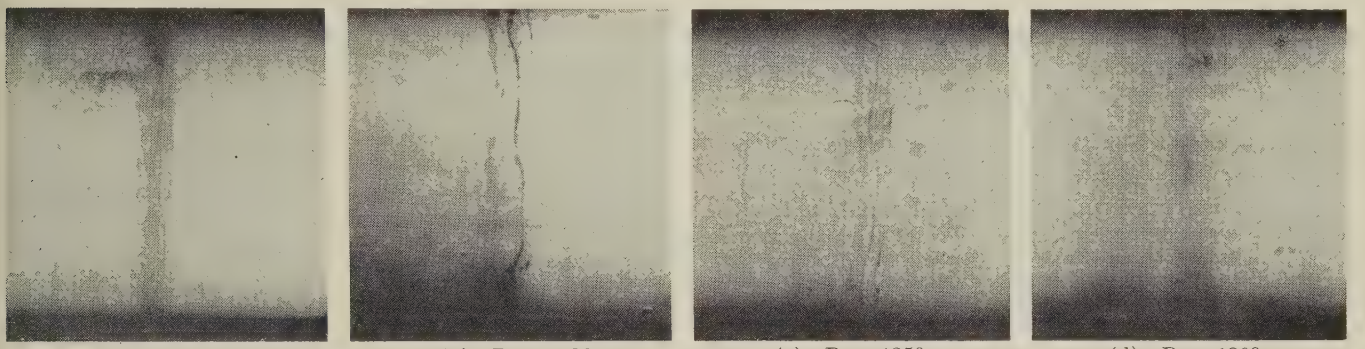


Fig. 5. Sectional view of Taylor vortices

at the core of the vortices. The air rings thus formed were not uniformly spaced. They were closer together at the section where the radial component of the flow was outward (see Fig. 4) and further apart where the radial component of the flow was inward. Photographs taken from directly above the annulus with only a thin horizontal section of the flow lighted (Fig. 5) showed that the cores of the vortices were also off center in the radial direction, being closer to the outer cylinder than they were to the inner cylinder. By scaling distances on this latter photograph, which was taken looking through a water box to eliminate optical distortion from the curvature of the outer cylinder, the location of the centers of the vortices was found to be $0.71b$ from the inner cylinder, and $0.33b$ from the dividing planes between the vortices on the side where the flow was radially outward. Although the flow shown in these figures contains a large number of air bubbles and is therefore nonhomogeneous, the same pattern was observed with fewer air bubbles, and is believed to be equally representative of a homogeneous liquid flow.

At the time this study was begun, no information was found from earlier studies to indicate the type of flow that occurs in Taylor vortices when they first form, that is, whether it is laminar or turbulent. The possibility of laminar flow was anticipated by Stuart when he wrote in a paper on hydrodynamic stability, "It does not follow that instability leads to turbulent motion, because another (possibly more complex) form of laminar motion may result" (*5*). The correctness of Stuart's prediction was shown by injecting dyed fluid into vortices and observing whether turbulence was present (*6*). Photographs of the dye pattern for different cylinder speeds (Fig. 6) showed the transition from laminar to turbulent flow. At the lowest Reynolds number the flow was laminar, and the thread of dye circulated without appreciable distortion other than a gradual spreading through the action of shear. At higher values of Reynolds number, the stream of dye was distorted and diffused, not only within one vortex but also into adjacent vortices. The exact value of the critical Reynolds number was difficult to determine, but for the case shown, the approximate value is $R_c = 1050$. Similar observations were made with inner cylinders for which b/r_1 values were 2.67 and 0.833. The critical Reynolds numbers for these were 800 and 950, respectively. Further comments on transition will be made in the following section.

Next attention was turned to the problem of measuring the strength of Taylor vortices. Two different methods were used. In the first, the special spinner described above was mounted so as to be in the very center of a vortex, thus indicating directly the rotational speed of fluid in the center of the vortex. This angular velocity was taken as a measure of vortex strength. In the second method, moving pictures were taken of neutrally bouyant particles of gilsonite suspended in the flow. The component of velocity due to the vortex motion was then determined from a graph of successive positions of particles in the flow. Agreement was obtained between the two methods of measurement.



(a) R = 920 (b) R = 1130 (c) R = 1250 (d) R = 1860

Fig. 6. Transition from laminar to turbulent vortices for $b/r_1 = 0.33$

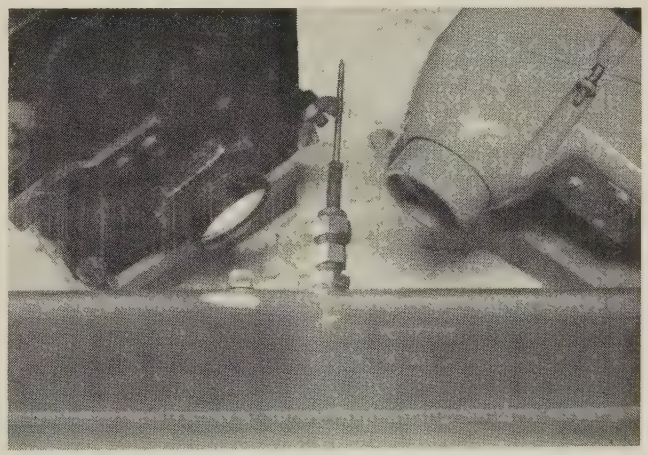


Fig. 7. Instruments for measuring spinner speed

The method of observing the spinner speed is shown in Fig. 7. A light beam (upper left) was reflected off the vanes of the spinner into a photoelectric cell (upper right). The output of the photocell was then amplified and put into a strip-chart recorder. For each quarter-turn of the spinner a pip was produced on the chart and from the spacing of these the speed of the spinner was determined. Difficulty was encountered in the first spinner measurements because a vortex would not remain in place when the spinner was located inside it. The retardation of the flow due to the resistance of the spinner made the fluid at this section to move radially outward, and this caused the vortices to form symmetrically on either side. However, by introducing a small axial flow the vortices were forced to pass across the spinner while a continuous record of the spinner rotation was obtained. The maximum spinner speed was taken as the rotational speed of the fluid in the core of the Taylor vortices.

To generalize the results of measurements of flow of widely differing viscosities, a Reynolds number was calculated for the vortex based on the angular speed w of fluid in the core of the vortex, or specifically, $R_v = r_1 w_v b/\nu$. The variation of this vortex Reynolds number with the Reynolds number of the primary flow between the cylinders is shown in Fig. 8 for the three relative annular widths tested. The slope of each curve is equal to the ratio of the angular velocity of the fluid in the vortex to the angular velocity of the inner cylinder and should be a constant within the range in which the flows are dynamically similar. Straight lines passing through the critical values of the Reynolds number of the primary flow given by Taylor's theory and lying approximately tangent to curves through the experimental points are shown. At low values of the primary Reynolds number, computed values of $w_v r_1 b/\nu$ generally lie below the straight lines, probably because the vortex motion is retarded due to viscous resistance to flow through the spinner. Also, a moderate amount of scatter of the results was obtained despite repeated attempts to hold conditions of observation constant.

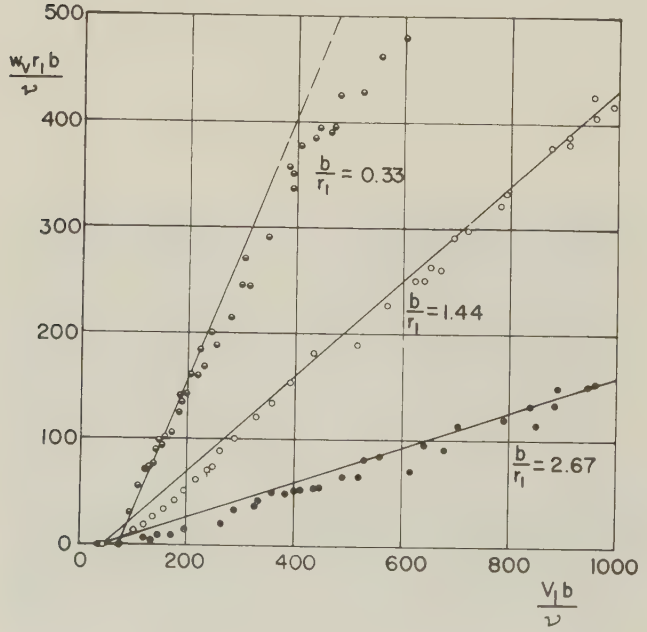


Fig. 8. Strength of Taylor vortices

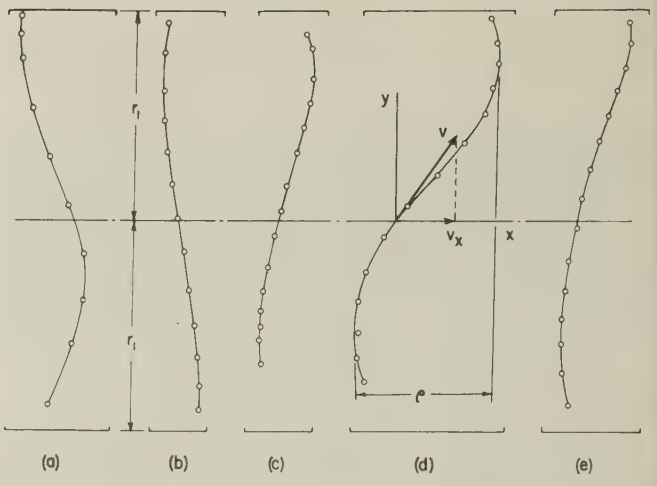


Fig. 9. Paths of gilsonite particles taken from movie film

Thus, a check by another method of measurement was deemed essential.

In the photographic method of measurement, particles of gilsonite, not exceeding $^1/_{16}$ in. diam. were mixed in a glycerol-water solution of approximately the same specific gravity, and their movement within Taylor vortices was recorded on a movie film taken at 16 frames per sec. By projecting this film one frame at a time onto a wall chart, and maintaining a pair of reference points in the picture at fixed points on the chart, the paths of individual particles could be plotted, as shown in Fig. 9. Next the motion of each particle was analyzed to obtain the rotational component of the velocity and the radius of the path of the particle in the vortex. The analysis was complicated by the three-dimensional nature of the flow. However, the mean angular velocity of a particle due solely to the vortex flow was obtained from the average radius of the path of the particle, ρ, and the x component of the velocity (see Fig. 9) of the particle as it passed across the midsection of the vortex. That is,

$$w_v = \frac{v_x}{\rho}$$

in which

w_v = the angular velocity in the vortex in radians per second
v_x = the axial component of the velocity in the vortex in feet per second
ρ = the average radius of the path of the particles in the vortex in feet

Only particles following a complete crossing from one side of the vortex to the other were selected and, furthermore, only those which also passed across the middle of the vortex near $y = 0$ could be used, since at this section the velocity in the z direction was essentially zero. The x component of the velocity was calculated from the ratio of the distance between successive positions of a particle, and the increment of time between exposures of the film. Particles moving along different radii within the vortices were observed, and a dimensionless diagram of the velocity distribution within the vortices was prepared, as shown in Fig. 10. Unexpectedly the velocity was found to increase linearly with radius so that the angular velocity in the core of the vortex could be easily calculated. From the slope of the velocity distribution curve

$$\left(\frac{v_x}{V_1 - V_c}\right)\left(\frac{b}{\rho}\right) = 0.66$$

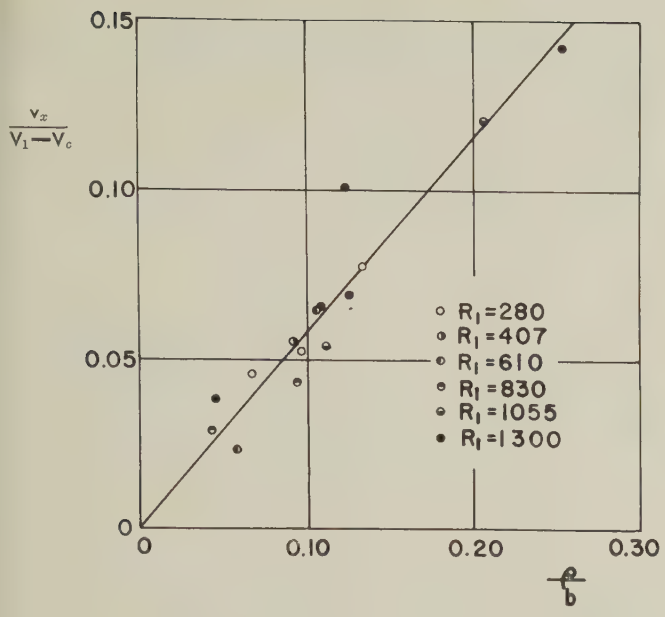


Fig. 10. Velocity distribution in vortex for $b/r_1 = 1.44$

a corresponding value of the ratio of the Reynolds number of the vortex flow to that of the primary flow was computed as follows:

$$\frac{R_v}{R_1 - R_c} = \frac{w_v r_1 b/\nu}{(V_1 - V_c)b/\nu} = \left(\frac{v_x}{V_1 - V_c}\right)\left(\frac{r_1}{b}\right)\left(\frac{b}{\rho}\right)^{-1} = \frac{0.66}{1.44} = 0.458$$

This result from the photographic method of measuring the strength of Taylor vortices agrees with the straight line shown in Fig. 8 for $b/r_1 = 1.44$ and provides a check on the results of the much simpler spinner method of measurement.

Once the point of transition to turbulent flow was reached, measurements of vortex strength became more erratic. The vortices often became unstable and wavered back and forth so that the position of the spinner could not be maintained in the center of a vortex. However, a tendency for the vortex to reach a maximum strength and then decrease with increasing speed of the inner cylinder was noted. Apparently the action of turbulence limited the development of vortices and, for flow at high Reynolds numbers, disrupted the vortices entirely.

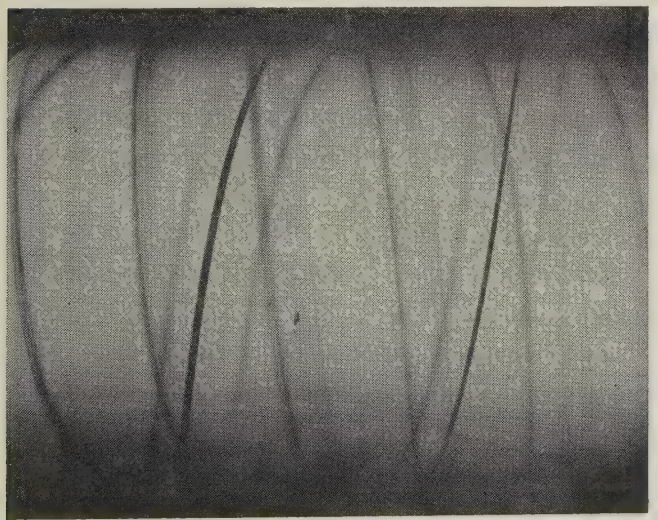


Fig. 11. Laminar flow in an annulus with axial discharge and rotation of inner cylinder

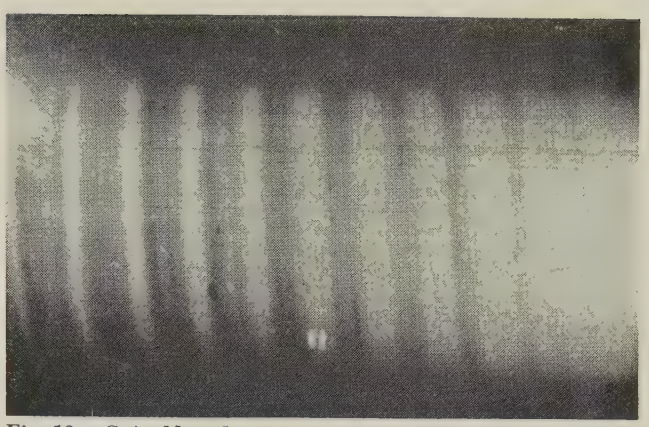


Fig. 12. Spiral band on inner cylinder before onset of Taylor vortices

EFFECTS OF ADDING AN AXIAL DISCHARGE

Addition of a small axial flow between the cylinders with rotational speeds below the critical for onset of Taylor vortices resulted in a motion which appeared to be a simple superposition of the two flows. The rotational component of the velocity was a maximum at the inner cylinder and decreased outward to zero at the outer cylinder, whereas the axial component of the velocity was zero at both walls and a maximum in the center of the annulus. The streamlines which resulted from this motion are shown very clearly in a 17-min. sound movie taken as part of this study and available on loan from the Kimberly-Clark Corp., Fluid Mechanics Laboratory, Neenah, Wis. An example of one such streamline, one near the wall of the outer cylinder, is shown in Fig. 11 as the darkest line of dye crossing the picture several times diagonally from lower left to upper right. The slope of this streamline indicates the ratio of the magnitude of the rotational and axial component of the velocity. In the background of this picture, and also in Fig. 12, another series of bands is seen having approximately half the spacing of successive loops of the dark streamline. These bands are located very close to the inner cylinder and rotate with the inner cylinder. Hence, they are not streamlines. With an increase in the axial flow , the spacing of these bands also increases. The reason for their formation is not known at the present time, but their occurrence suggests that there is some sort of secondary motion very close to the inner cylinder.

After the onset of Taylor vortices, the vortex pattern is essentially uneffected by a small axial flow, but is transported along with it. Near the walls of the cylinders the axial flow is either to the right or the left, depending on the sense of the rotation in the vortex passing the particular point at the time. The flow at the boundaries is controlled by the vortex motion. With larger axial flows, the motion of the fluid at the walls is no longer controlled by the vortices. Rather boundary layers develop on the cylinders, as illustrated by the dark streamline in Fig. 13. The vortices are confined to the space between the boundary layers on the cylinder walls and, as before, move along with the axial flow. Thus the nature of the flow close to the walls of the cylinders was found to depend on the relative magni-

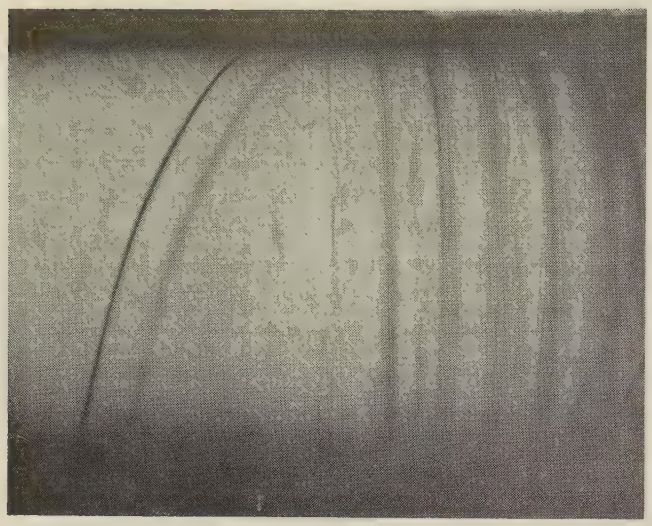


Fig. 13. Taylor vortices inside laminar boundary layers

tudes of the axial discharge and the strength of the Taylor vortex motion. More information on this aspect on the flow between rotating cylinders is needed particularly in connection with problems of heat transfer from the surfaces of the cylinders.

After this study had been started, a report on a related investigation was published by Kaye and Elgar (*7*). In this work the point of change from laminar flow to laminar Taylor vortex motion and the transition from laminar to turbulent flow with or without Taylor vortex motion were determined for two annular widths with air as the fluid medium. A composite of the results, showing also data from earlier work by Cornish (*8*) and Fage (*9*) is reproduced from the report by Kaye and Elgar as Fig. 14 herein. The ordinate in this graph is the Reynolds number of the axial flow and the abscissa is a function of R_1 and b/r_1 (w is the angular velocity of the inner cylinder, r_m is the mean of the radius of the cylinders, and F_g is a geometric factor depending on b/r_m). Thus all combinations of cylinder speeds and axial flows are represented on this diagram. The critical Reynolds numbers for transition from laminar Taylor vortices to turbulent Taylor vortices with zero axial flow were noted in the section on motion within Taylor vortices and these may be used to supplement the results shown

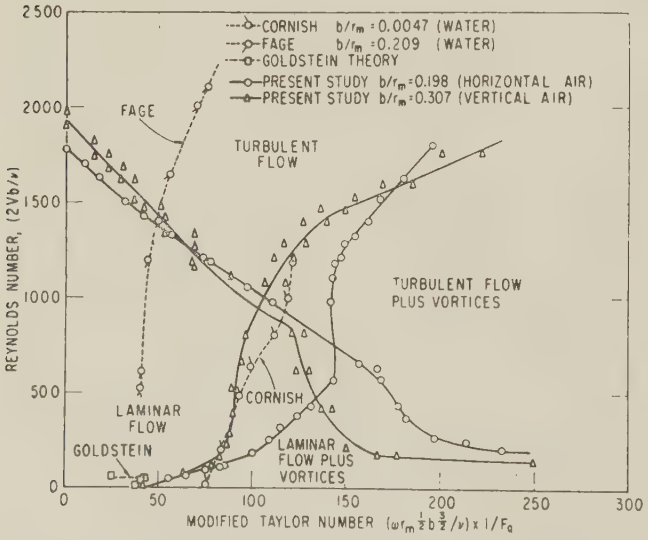


Fig. 14. Summary of data on four regimes of flow from paper by Kaye and Elgar

in Fig. 14. Values of the modified Taylor number for observed critical flows have been calculated using the equation given by Kaye and Elgar for the geometric factor

$$F_g = \frac{\pi^2}{41.2}\left(1 - \frac{b}{2r_m}\right)^{-1} P^{-1/2}$$

in which

$$P = 0.0571\left[1 - 0.652\left(\frac{b/r_m}{1 = (b/2r_m)}\right)\right] + 0.00056\left[1 - \frac{0.652(b/r_m)}{1 - (b/2r_m)}\right]^{-1}$$

For values of b/r_1 greater than 1.5 the modified Taylor number becomes imaginary. This parameter originated in a theoretical analysis applicable to narrow annuli only and loses meaning when applied to wide annuli.

The values of critical Taylor number for zero axial flow, given in Table I, lie off the graph in Fig. 14 but serve to indicate the end point of the curves for transition as the Reynolds number approaches zero. The critical condition for transition from laminar to turbulent vortices apparently depends on the relative annular width and is markedly effected by even small axial flows.

CONCLUSIONS

The characteristics of fully established Taylor vortices in the annuli between concentric cylinders, the inner rotating and the outer one stationary, have been studied. Also the effects of adding an axial flow have been observed.

A stable laminar secondary flow in the form of Taylor vortices has been clearly shown to occur. These vortices were found to be markedly unsymmetrical, the core of each vortex lying closer to the outer cylinder than to the inner, and nearer to the section where the radial component of flow was outward than to the dividing plane on the other side along which the vortex flow was inward.

For flow at Reynolds numbers up to the critical for transition to turbulent vortex motion, the strength of the vortices was found to increase in direct proportion to the increase in speed of the inner cylinder for three different annular widths tested. With development of turbulence in the flow, a tendency was noted for the strength of the Taylor vortices to reach a maximum and then to decrease with increasing cylinder speed.

Addition of an axial flow was found to give rise to the following types of motion:

1. Laminar flow (with possible local instability close to the inner cylinder),
2. Laminar Taylor vortex flow in the space between boundary layers due to the axial flow,
3. Fully developed laminar Taylor vortex flow (completely filling the annulus),

Table I

$\frac{b}{r_1}$	$\frac{b}{r_m}$	R_c	*Modified Taylor no.* $\left(\frac{\omega r_m^{1/2} b^{3/2}}{\nu} \times \frac{1}{F_g}\right)$
0.33	0.286	1050	500
0.833	0.558	950	350
2.67	1.145	800	...

4. Turbulent Taylor vortex flow between boundary layers due to the axial flow,

5. Fully developed turbulent Taylor vortex flow.

Lastly, the results of observations of the point of transition from laminar to turbulent Taylor vortices with zero axial flow are presented to supplement the recently published data of Kaye and Elgar for transition with axial flows.

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Secondary Flow Phenomena in Nonlinear Fluids

J. L. ERICKSEN

Flow of nonlinear fluids sometimes differs qualitatively from that predicted by the classical linear theory of viscous fluids. In general, some part of the flow is similar to that predicted by linear theory. The remainder, which has no classical analog, is called a secondary flow. This is described and theoretical and experimental work bearing on this are correlated.

STRESSES

Consider the simple shearing motion with velocity components

$$u = \dot{\gamma}y,\ v = w = 0,\ \dot{\gamma} = \text{const.} \tag{1}$$

A priori, as many as six stresses might be involved in maintaining this. Three of these may be taken as the stresses σ_x, σ_y and σ_z acting normal to the planes $x = \text{const.}$, $y = \text{const.}$, and $z = \text{const.}$, respectively. The others may be taken as any three independent shear stresses acting on these planes. We choose the shear stress τ acting on the planes $y = \text{const.}$ in the direction together with the two shear stresses τ_{xz} and τ_{yz} acting on the plane of shear ($z = \text{const.}$) in the x and y directions. For incompressible materials, superimposing a hydrostatic pressure does not effect the motion, but changes σ_x, σ_y, and σ_z by equal amounts. If the motion is maintained for a sufficiently long time to permit any relaxation effects to die out, it seems reasonable to assume that, in fluids, the normal stress differences and shear stresses will be determined by $\dot{\gamma}$,

$$\left.\begin{aligned} \sigma_x - \sigma_z &= f(\dot{\gamma}) \\ \sigma_y - \sigma_z &= g(\dot{\gamma}) \\ \tau &= h(\dot{\gamma}) \\ \tau_{xz} &= k(\dot{\gamma}) \\ \tau_{yz} &= m(\dot{\gamma}) \end{aligned}\right\} \tag{2}$$

In a fluid, we would expect that the shear stress τ required to produce a given rate of shear to the right ($\dot{\gamma} > 0$) will be numerically equal but oppositely directed to that producing a rate of shear of the same amount but to the left ($\dot{\gamma} < 0$), which means that h should be an odd function of $\dot{\gamma}$. Similar symmetry considerations indicate that $\sigma_x - \sigma_z$ and $\sigma_y - \sigma_z$ should be even functions of $\dot{\gamma}$ while τ_{xz} and τ_{yz} should vanish. We may then write

$$\left.\begin{aligned} \sigma_x - \sigma_z &= \alpha(\dot{\gamma}^2)\dot{\gamma}^2 \\ \sigma_y - \sigma_z &= \beta(\dot{\gamma}^2)\dot{\gamma}^2 \\ \tau &= \eta(\dot{\gamma}^2)\dot{\gamma} \\ \tau_{xz} &= \tau_{yz} = 0 \end{aligned}\right\} \tag{3}$$

η being the apparent viscosity. The classical linear theory gives

$$\alpha = \beta = 0, \qquad \eta = \text{const.} \tag{4}$$

Fluids described by (4) are said to be Newtonian, others being called non-Newtonian. Nonzero values of α or β are encountered in fluids exhibiting the Weissenberg effect, i.e., fluids which climb up a rotating stir rod inserted in them. This climbing motion is perhaps the best known example of secondary flow. In high polymer solutions, available data suggest that $(\sigma_x - \sigma_z)/\dot{\gamma}^2$ and $(\sigma_y - \sigma_z)/\dot{\gamma}^2$ remain finite as $\dot{\gamma} \to 0$, though this point has not been examined critically. It is also theoretically convenient to separate out a factor $\dot{\gamma}^2$ from the normal stress differences to put the relations (3) in tensor or matrix form. One such form is

$$\underset{\sim}{t} = -p\underset{\sim}{1} + \eta(\dot{\gamma}^2)\underset{\sim}{A} + \alpha(\dot{\gamma}^2)(\underset{\sim}{A}^2 - \underset{\sim}{B}) + \beta(\dot{\gamma}^2)\underset{\sim}{B}$$
$$2\dot{\gamma}^2 = \text{trace } \underset{\sim}{A}^2 \tag{5}$$

where $\underset{\sim}{t}$ is the stress matrix, p is an arbitrary isotropic pressure, $^1/_2\underset{\sim}{A}$ is the rate of strain tensor commonly used in fluid mechanics and $\underset{\sim}{B}$ is a strain acceleration tensor. In terms of Cartesian velocity components v_i,

$$A_{ij} = v_{i,j} + v_{j,i}$$

$$2B_{ij} = \frac{\partial A_{ij}}{\partial t} + A_{ij,k}\,v_k + A_{im}v_{m,j} + A_{jm}v_{m,i}$$

These relations are used in obtaining results to be described, but we avoid the rather complicated calculations which involve them.

For polyisobutylene solutions, Markovitz (*1*) has

J. L. Ericksen, Associate Professor of Theoretical Mechanics, Mechanical Engineering Dept., The Johns Hopkins University, Baltimore, Md.

used relations equivalent to (5) in correlating and summarizing available experimental data for polyisobutylene solutions. For these, the empirical relation

$$\frac{d \ln \eta}{d \ln \dot{\gamma}} = -0.12 \ln \dot{\gamma} + \text{const.} \qquad (6)$$

holds rather accurately. As yet, there are unresolved discrepancies in the meager data obtained from different kinds of normal stress measurements. Data obtained by Roberts (*2*) from cone and plate viscometers suggest that

$$\beta = 0, \alpha \neq 0 \quad \text{or} \quad \sigma_x \neq \sigma_y = \sigma_z \qquad (7)$$

Data obtained from torsional shearing between circular plates, combined with that obtained from shearing between coaxial cylinders are in contradiction with (7). Markovitz (*1*) points out that these data suggest that

$$\alpha = 2\beta \quad \text{or} \quad \sigma_x + \sigma_z - 2\sigma_y = 0. \qquad (8)$$

As discussed by Criminale, Ericksen, and Filbey (*3*), all data suggest that, as a rough approximation for $\dot{\gamma}$ neither too small nor too large,

$$\alpha = a/|\dot{\gamma}| = a/\sqrt{\dot{\gamma}^2}, \quad \beta = b/|\dot{\gamma}| = b/\sqrt{\dot{\gamma}^2}, \qquad (9)$$

where a and b are constants. In a 15% solution of polyisobutylene in decalin, data published by Philippoff (*4*) indicate that $\sigma_x - \sigma_y$ and τ are nearly equal for $\dot{\gamma} \approx 0.3$ sec.$^{-1}$. Thus the normal stress differences are hardly negligible. One possible implication of practical importance is that a Reynolds number calculation designed to predict when laminar flow will become unstable may be quite misleading. In polymer melts, observed instability occurs at Reynolds numbers well below those at which turbulence occurs in Newtonian fluids. This instability, the so-called melt fracture phenomenon, is thought by some to be associated more closely with normal stress differences than with the shear stress τ. The phenomenon is discussed by Tordella (*5*).

It should be noted that, according to general theories of continua such as those discussed by Rivlin and Ericksen (*6*), Green and Rivlin (*7*) and Noll (*8*), the relations (5) are applicable only to very special types of flow. They do apply to some of the more common shear flows such as Poiseuille flow and Couette flow, as is rather clear from Rivlin's (*9*) general solutions. Known flows to which they apply are listed by Ericksen (*10*).

COUETTE FLOW

By Couette flow, we mean a motion of the type

$$u = -yf(r), v = xf(r), w = 0, r^2 = x^2 + y^2 \qquad (10)$$

That is, the particles remain in planes z = const., traveling in circles with centers at the z-axis. Each particle travels with constant speed, the value of which varies from particle to particle. Thus this represents some approximation to the flow occurring near a rotating stir rod inserted in a container of fluid. One can use (5) to calculate corresponding stresses, which may then be inserted in the equations of equilibrium. This yields the equation

$$f'\eta(r^2f'^2) = k/r^3, \quad k = \text{const.} \qquad (11)$$

for determining $f(r)$ and a determination of p. Details are discussed by Rivlin (*9*). Neglecting inertia and gravity, the normal stress acting on a surface z = const., is found to be given by

$$N = -r^2f'^2\beta(r^2f'^2) + \int [\alpha(r^2f'^2) - \beta(r^2f'^2)]rf'^2\, dr. \qquad (12)$$

In general, it is impossible for such a surface to be open to the atmosphere, for this would require N, which generally varies with r, to be equal to atmospheric pressure, which does not. It is conceivable that a curved free surface might be possible, but this can similarly be disposed of. Thus, a secondary flow must occur for a free surface to exist. If the force $N(r)$ required to maintain the flow (10) were removed or replaced by a constant force, the fluid should move vertically, the vertical motion depending on r. If N increases with r, upward motion should decrease with r and vice versa. In a qualitative way, this explains the tendency of some fluids to climb the stir rod. In some situations, the secondary flow is very pronounced. When

$$N' = \frac{dN}{dr} = -[r^2f'^2\beta(r^2f'^2)]' + [\alpha(r^2f'^2) - \beta(r^2f'^2)]rf'^2 \equiv 0,$$

the secondary flow need not occur. From (4), this is the case for Newtonian fluids. It is very unlikely that it holds at all rates of rotation of the stir rod for any real fluid except those for which $\alpha = \beta = 0$, since one can show that it requires that, in simple shear

$$\alpha\dot{\gamma}^2 = \beta\dot{\gamma}^2 = \text{const.} = \sigma_y - \sigma_z = \sigma_x - \sigma_z \qquad (13)$$

For most real fluids, $\sigma_x = \sigma_y = \sigma_z$ when $\dot{\gamma} = 0$, which is consistent with (13) only if $\alpha = \beta = 0$.

The experiment indicated here is thus a simple and reliable test for determining whether α or β is nonzero for a given fluid. When $\alpha = \beta = 0$, inertia may cause a secondary flow, but this always tends to depress rather than raise the surface near the stir rod relative to that farther away.

Further discussion of and references to work on Couette flow of nonlinear fluids are given by Truesdell (*11*), §72.

We have been informed that there are indications that α and β are not both zero in fiber suspensions, but have seen no publications concerning this.

FLOW IN NONCIRCULAR PIPES

Classical fluid mechanics predicts that, in the case of laminar flow through an infinitely long circular or noncircular pipe, each particle will move in a straight line with constant speed. The speed will of course vary from particle to particle in general. For non-Newtonian fluids, theoretical investigations by Ericksen (*12*), Green and Rivlin (*13*), Langlois (*14*), Stone (*15*), and Criminale, Ericksen, and Filbey (*3*) have shown that this simple type of motion is mathematically impossible unless the fluid is such that the ratio

$$R \equiv (\sigma_y - \sigma_z)/\tau\dot{\gamma} = \beta/\eta \qquad (14)$$

is independent of $\dot{\gamma}$ in simple shearing motion. For circular pipes, the simple rectilinear motion is possible quite generally, as is indicated by the general solution for this case given by Rivlin (*9*), for noncircular pipes, a steady motion is generally mathematically possible. It consists of a simple rectilinear motion on which is superimposed a vortexlike secondary motion in cross-sectional planes. That is the particles follow spiral

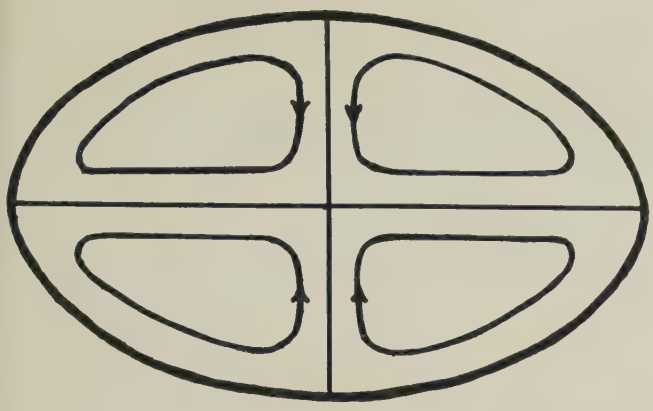

Fig. 1

paths as they traverse the pipe. Illustrative solutions for elliptical pipes are given by Green and Rivlin (*13*) and Langlois (*14*). Various secondary flow patterns can occur, depending on the values of material moduli. One of the simpler possibilities is illustrated in Fig. 1.

From one viewpoint, it is not surprising that these secondary flows should occur. In the "time averaged" motion of Newtonian fluids in turbulent flow through noncircular pipes, such secondary flows are observed. A description of these is given by Schlichting (*16*), Ch. XX. As is discussed by Rivlin (*17*), there is at least a loose analogy between turbulent flow of Newtonian fluids and laminar flow of non-Newtonian fluids.

In principle, we could estimate whether secondary flows should occur by using experimental data to calculate R, hence to determine whether R is constant. For polyisobutylene solutions, (7) implies that $R = 0$, hence that secondary flows need not occur. On the other hand, (6), (8), and (9) imply that R is not constant, as is noted by Criminale, *et al.* (*3*). If this is correct, secondary flows must occur. Hence, available data do not settle the question for these fluids.

It would of course be more satisfactory to observe laminar flow of non-Newtonian fluids in noncircular pipes and thus determine directly whether secondary flows occur. G. Hand has made some preliminary attempts to do this, finding nonreproducible secondary flows in some fluids, no secondary flows in other. Further experiments covering a wider range of rates of shear are planned to settle the question more conclusively. If it turns out that secondary flows are not observed here, it would be almost certain that (7) is correct.

The possible occurrence of these secondary flows suggests the interesting possibility of using noncircular pipes to produce a mixing action which may sometimes be desirable.

OTHER SECONDARY FLOWS

Secondary flow phenomena may occur in several other types of geometries. In the flow between concentric right circular cones in relative rotation, one of which may degenerate to a flat plate, it is noted by Ericksen (*10*) that secondary flows must theoretically occur unless the stresses in simple shear satisfy

$$\sigma_x + \sigma_y - 2\sigma_z = \text{const.} \qquad (15)$$

This assumes that the effects of inertia are negligible, so that, classically, particles on a cone concentric with the boundaries move as a rigid body. Equation (15) holds for any fluid for which $\alpha = \beta = 0$. As is rather clear from (6), (7), and (8) it does not for polyisobutylene. A secondary flow can also arise as an inertial effect. Estimates of the relative importance of the two types of secondary flows are lacking, it being still uncertain whether they differ qualitatively. Cone and plate or biconical viscometers have this geometry. Experiments of Roberts (*2*) indicate that one can work in a range where both secondary flows are too weak to be observed. For some fluids, he notes that complicated secondary flows occur rather suddenly as the rate of rotation is increased. To avoid misinterpreting data from such instruments, it is thus wise to provide for direct observation of the flow pattern, at least until these secondary flows are better understood theoretically.

Theoretical analyses indicating noninertial secondary flow in the flow between concentric spheres in relative rotation and in flow in a plane wedge are given by Langlois (*18*) (*14*). Again, these do not occur in fluids with $\alpha = \beta = 0$. For fluids for which α or β is nonzero, the nature of these flows depends on certain material constants. For high polymers, it appears that the secondary motion would be in a direction opposite to that produced by inertia. There seems to be no experimental information concerning these flows.

CONCLUSIONS

We have attempted to give a birdseye view of present knowledge of secondary flow phenomena in nonlinear fluids. Perhaps the major importance of such investigations is that they give some insight into the influence of the normal stresses which are so frequently ignored in analyses of nonlinear fluids. When experiments such as that discussed in Couette flow indicate significant normal stress differences in the fluid of interest this practice seems unsafe in general, though it may not lead to error in special situations. Much more work on normal stresses needs to be done. For example, virtually nothing concrete is known about the influence of these on turbulence.

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Two New Methods for Determining Loadings of Colloidal Silica on Paper Surfaces

G. J. McEWAN, R. P. BEIMLER, E. F. KAELBLE, and T. A. TAULLI

Recent increased interest in the application of colloidal silica for imparting nonskid properties to paper surfaces has pointed up the need for methods of determining silica loadings. Two methods have been developed for this purpose, a colorimetric method and an x-ray spectrographic method. Sensitivity, speed and accuracy are features of these methods.

Colloidal silica is used in both the multiwall bag and corrugated box industry for imparting skid resistance to paper and paperboard containers. This use is described in U. S. pat. 2,643,048, issued in June of 1953 and assigned to Monsanto Chemical Co. It is generally accepted as an effective yet economical answer to the problem of the slipperiness of paper surfaces if it is applied uniformly and in sufficient amounts. One difficulty which becomes evident when such a material is applied commercially is the lack of a rapid method of determining the amount and the uniformity of the added silica. The problem is compounded by the very small amount of material used per unit area and the inherent variability of the treated substrate. Colloidal silica (e.g. SYTON* P, Monsanto's colloidal silica) is frequently added in amounts of 0.1 lb. of solids per 1000 sq. ft. of treated surface. On a weight percentage basis this would cover a range of about 0.5 to less than 0.1% depending on whether the colloidal silica was applied to multiwall bag paper or to corrugated board.

Methods which have been used to gather loading information include:

1. Ashing (wet or dry), or ashing followed by hydrofluoric acid treatments to volatilize the silica portion of the ash. These analyses are time consuming and may be subject to many errors of technique.

2. Obtaining an over-all average at the end of a particular run by measuring the amount and concentration of solution used and by knowing the area of the treated surface. This procedure gives only an average usage figure for the particular run in question and gives no information on the uniformity of application. In addition, it cannot be conveniently used on runs of short duration as for example when testing several application variables; it cannot be used to check a particular sample which may be in question; and it is, at best, an indirect measurement of what appeared to be applied.

3. Making performance checks while the run is in progress. While this procedure has much to recommend it, it may give erroneous results since slip-resistant performance is very dependent on the testing conditions and the history of the sample being tested. That board which performs satisfactorily in the mill under prevailing conditions of humidity and temperature may not perform as well in a customer's plant under different exposure conditions. Performance tests alone would not answer the question of whether a certain amount of silica was applied, as when following a specification.

The ultimate answer would be a device which could be used in a plant or mill while the application was being made and which would directly give the amount of silica applied. Unfortunately we know of no such device. However, presented below are two methods which have recently been developed by us. These methods offer relatively simple, fast procedures for determining the amount of silica on a paper surface and so may serve until such a device as described above is perfected. In addition, the principles exemplified in these methods may aid in the development of such a device.

X-RAY SPECTROGRAPHIC METHOD

Background

This method is based on the phenomenon occurring when silica particles are bombarded with x-rays; the silicon atoms emit characteristic secondary radiations which can be collected and counted. The number of these secondary radiations is proportional to the amount of silica present.

It is unusual for x-ray spectrography to find use in measuring concentrations as low as that of silica on paper. Meaningful x-ray measurements are here possible, however, because x-ray spectrography sees essentially just the surface of a sample, and the silica is concentrated on the surface of the paper. Thus, although the absolute amount of silica is very small, the ratio of silica to paper at the surface is relatively high. Generation of secondary radiation occurs deeper in a sample, but the radiation is absorbed by the sample without being measured.

Instrumental Conditions

A standard Norelco x-ray spectrograph was used in this study. The primary x-ray beam was generated in a Philips FA-60 tungsten-target x-ray tube operated at 50 kv. and 45 ma. The secondary radiation emitted by the sample was monochromatized by reflection from an ethylene-diamine-*d*-tartrate analyzing crystal and collimated by passage through a 0.02-in. parallel plate collimator. Intensity measurements were made with a gas flow proportional counter set to measure the wavelength of the silicon K-alpha emission line (7.125 A). The time required to collect a predetermined number of counts was used as an index of intensity. The number of counts was varied to give counting times of 1 to 2 min.

G. J. McEwan, R. P. Beimler, E. F. Kaelble, and T. A. Taulli, Research Dept., Inorganic Chemicals Div., Monsanto Chemical Co., St. Louis, Mo.
* Syton: Reg. Trade Mark Monsanto Chemical Co.

Background intensity was determined by counting paper samples which contained no silica. An Atomic Instruments Co. pulse height analyzer was used to minimize background. The optical path of the spectrograph was purged with helium to eliminate absorption of the silicon radiation by air.

Preparation of Standard Curve

Pieces of multiwall bag paper were hand-sprayed as uniformly as possible with colloidal solutions of silica. To vary the amount of silica on the papers, several different concentrations were applied in from one to three spray passes. Two grades of paper were used, regular finish and extra rough finish.

A 5- by 8-in. portion of each paper was divided into 20 rectangular pieces. The intensity of the silicon K-alpha line was measured for each of the 20 pieces after which the silica content of the combined pieces was determined by ashing. Average x-ray intensity less background was plotted against pounds silica per 1000 sq. ft. paper. The resulting curve is shown in Fig. 1.

The fact that all points lie on the same curve indicates that within the limits used, the method is independent of paper grade, concentration of silica sprayed, and

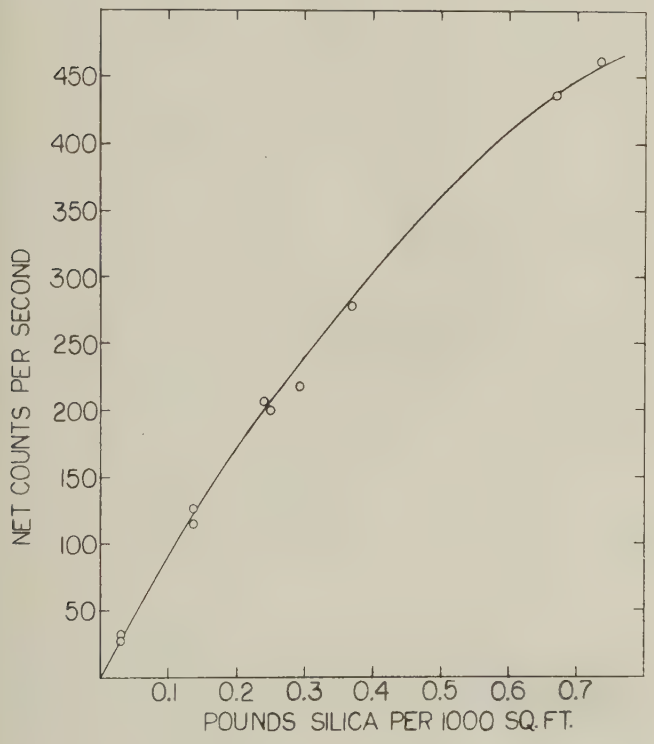

Fig. 1. Standard curve for x-ray spectrographic determination of silica on paper surfaces

number of spray passes. Deviation from linearity is commonly encountered in x-ray spectrographic standard curves covering a wide range of concentrations. In this case, it is caused by self-absorption of some of the silicon K-alpha radiation by the silica particles at the higher concentrations.

The curve presented in Fig. 1 is representative only of the conditions and equipment used in this investigation. It should be redetermined in all future applications.

Analysis of Samples

To determine the concentration of silica on a sample of paper, the silicon K-alpha intensity given by the paper is measured and corrected for background. The corresponding silica concentration is then read from the standard curve. The number of replicates of each sample to be run will depend upon the uniformity of the silica coating and/or upon the accuracy desired.

Discussion

As little as 0.01 lb. silica per 1000 sq. ft. paper can be determined by this method. At this level, only about 20γ of silica is exposed to the x-ray beam. On a weight per cent basis, this corresponds to 0.05% silica on bag paper and to 0.01% on corrugated board. This is unusually high sensitivity for an x-ray spectrographic determination of a light element. As was stated above, the explanation is that the silica is concentrated on the surface of the paper, the only portion of the sample to which the x-ray spectrograph is sensitive.

Accuracy of the method depends upon the accuracy of the silica content of the standards used to prepare the standard curve. Using the procedure described here, accuracy of at least 10% relative was obtained. The method is rapid, requiring only 3 to 5 min. per analysis.

MOLYBDENUM YELLOW—COLORIMETRIC METHOD

Background

The general procedure for determining silica by the molybdenum yellow technique can be found in almost any standard quantitative analysis textbook. While numerous adaptations of this colorimetric method have been reported in the literature, none has been directly applied to the analysis of silica on paper.

Principle

The sample is digested with boiling sodium hydroxide solution to dissolve and convert the silica to a form suitable for colorimetric determination. Leachings from the resulting pulp mass are treated with an acidic ammonium molybdate solution to produce a yellow-colored solution. Absorbancy measurements on the colored solutions are then correlated with silica concentrations from the previously prepared calibration curve.

Reagents

Sodium Hydroxide Solution—0.01 *N*, 2.5 *N*.

Sulphuric Acid Solution—0.3 *N*, 2.0 *N*.

Ammonium Molybdate Solution—5%. Five grams of A. R. grade ammonium molybdate tetrahydrate are dissolved in 80 ml. of warm distilled water. The solution is cooled; 2.8 ml. concentrated sulphuric acid is added, and the solution diluted to 100 ml. in a volumetric flask.

Standard Silica Solution—(0.1 mg. SiO_2 per ml.). Transfer an equivalent of 0.1000 g. (previously assayed) SiO_2 to a platinum crucible; add 10 ml. distilled water and 0.5 g. NaOH and heat gently to dissolve the silica. Cool contents to room temperature and transfer the solution quantitatively to a 1-liter volumetric flask. Dilute to the mark with distilled water.

Preparation of Standard Curve

Pipet 5, 10, and 15 ml. of standard silica solution (0.1 mg. SiO_2/ml.) to a stainless steel cup, add 10 ml. 0.01 *N* NaOH and heat just to boiling. Cool contents to approximately 25°C. and add sufficient distilled water to give a total volume of 70 ml. Using a pH meter, adjust the pH of the solution to 1.4 ± 0.05 with 0.3 *N*

H_2SO_4. Add, by pipet, 7.5 ml. of ammonium molybdate solution. Swirl to mix contents. Quantitatively transfer solution to a 100-ml. volumetric flask, dilute to mark with distilled water and mix well. Transfer a portion of the solution to a 1-cm. matched Corex cell and

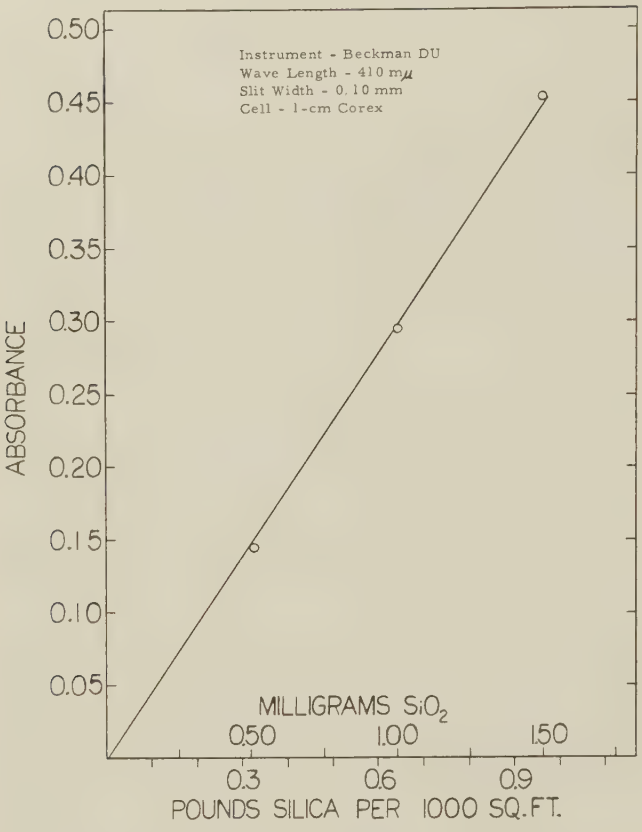

Fig. 2. Standard curve for colorimetric determination of silica on paper

read the absorbancy of the solution at 410 mμ using a 0.10 mm. slit width and a Beckman Model DU spectrophotometer or an equivalent instrument. Distilled water is used as the reference solution.

Since the color intensity of the solution will decrease on standing, absorbancy measurements should be made within 5 to 10 min. after the molybdate solution is added. The absorbancy of a reagent blank, containing all reagents with the exception of the standard silica solution, should be deducted from the absorbancy of all silica-containing solutions. A standard curve is then prepared by plotting the weight in milligrams of SiO_2 versus net absorbancy units (Fig. 2). The relationship was found to be linear. For convenience, this scale was converted to read pounds silica per 1000 sq. ft. (based on a 25 sq.-in. sample of paper).

Analysis of Sample

Cut a 25 sq.-in. sample from the paper (if corrugated board is being used, peal liner from board before cutting sample), weigh and place it in a 150-ml. stainless steel beaker containing 50 ml. of a 2.5 *N* NaOH solution. Boil for 5 min. with agitation (provided by a motor-driven, stainless-steel impeller). Add distilled water to keep the volume constant. Vacuum filter the resulting slurry through a polyethylene Buchner funnel and wash pulp with a small amount of distilled water. Remove paper pulp plus filter paper and repeat leaching steps two times using fresh portions of 2.5 *N* NaOH solutions. Transfer combined filtrates quantitatively to a 500-ml. volumetric flask. Wash all of the filtrate from the filter flask and stainless steel beaker with a stream of distilled water. Dilute to mark with distilled water and mix well. Pipet a 10-ml. aliquot from the 500-ml. flask to a stainless steel beaker and add about 20 ml. of distilled water. Using a pH meter, adjust the pH of the solution to 1.4 ± 0.05 with 0.3 *N* H_2SO_4 (use 2.0 *N* H_2SO_4 to bring pH to about 1.6). Adjust volume to 70 ± 5 ml. with distilled water. By pipet, add 7.5 ml. of ammonium molybdate solution, swirl to mix and quantitatively transfer to a 100-ml. volumetric flask. Dilute to mark with distilled water and mix well. Transfer a portion of the solution to a 1-cm. matched Corex cell and read the absorbancy of solution at 410 mμ (0.10-mm. slit width) *within 5 to 10 min.* after the molybdate solution was added. Use distilled water in the reference cell.

Determine a blank from a sample of paper before silica treatment (of the same weight and approximately same area) as directed above. Subtract the absorbancy of the blank from the absorbancy of the treated sample. From the prepared standard curve convert net absorbancy reading to equivalent milligrams SiO_2 and/or pounds silica per 1000 sq. ft.

Discussion

In preparing the standard curve, it was found necessary to compensate for the silica introduced by the reagents and for equipment contamination. In addition, correction is applied in the actual analysis of the sample to compensate for the yellow pigments extracted from the paper substrate. This is accomplished by analyzing a sample of the paper which has not been treated with silica. Based on known silica loadings in laboratory prepared samples, the accuracy of the method was found to be about 10% relative. Printed portions of paper should be avoided as they introduce added interference.

Since similar type instruments may be expected to give different readings for the same solution, a standard curve should be prepared with the particular instrument used.

COMPARISON OF THE TWO METHODS

From time to time some samples of treated paper or corrugated board have been analyzed for silica content by these two methods. The results of these cross checks are presented below:

Silica loading (pounds SiO_2/1000 sq. ft.)	
X-ray method	*Colorimetric method*
0.42	0.47
0.18	0.20
0.04	0.04
0.06	0.08

The x-ray method has the following advantages:

1. It is fast—an individual determination requires about 3 to 5 min.
2. It measures the silica on a small area and therefore can be used to determine uniformity of coating.
3. It is nondestructive, permitting subsequent analyses.

4. It measures only the effective silica, that is, the silica at the surface.

However, it has one rather serious drawback. The necessary equipment is expensive and bulky.

The colorimetric method has the following advantages:

1. Relatively inexpensive equipment, found in many laboratories, is used.
2. Replicate samples can be run concurrently.

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Alkaline Hydrolysis of Representative Palms

IRWIN A. PEARL, DONALD L. BEYER, and DAWN LASKOWSKI

The woody leaf stalks or petioles of 26 representative species of palms (family *Palmaceae*) were subjected to alkaline hydrolysis. The alkaline hydrolyzates were acidified and extracted with ether. The ether extracts were analyzed qualitatively by paper chromatography and quantitatively by paper chromatographic and spectrophotometric procedures for phenolic materials. In all species of palm examined, *p*-hydroxybenzoic acid was the chief product of alkaline hydrolysis. In addition, all palm species yielded vanillin, syringaldehyde, acetovanillone, acetosyringone, vanillic acid, and syringic acid when hydrolyzed with boiling alkaline solution. Other compounds, both known and unknown, were found in only a portion of the species examined. Some compounds were very specific to certain species, and may have taxonomic significance. The occurrence of these compounds, their qualitative and quantitative relationships, and their relation to other wood components are discussed.

DURING the course of our studies on the alkaline hydrolysis of representative hardwoods (*1*, *2*) we had occasion to submit trunk material from the cabbage palmetto (*Sabal palmetto*) to hydrolysis with boiling normal sodium hydroxide solution. The results obtained (see Table I) were unusual when compared with earlier results on the representative hardwoods. As in the hardwoods, vanillin, syringaldehyde, *p*-hydroxybenzaldehyde, vanillic acid, syringic acid, ferulic acid, and *p*-hydroxybenzoic acid were found in the alkaline hydrolyzate of the palmetto. In addition, acetosyringone and acetovanillone were found. The total ether extract produced upon alkaline hydrolysis was approximately 4% of the dry starting material, and of this over 80% was accounted for by *p*-hydroxybenzoic acid. Heretofore, *p*-hydroxybenzoic acid had been found in quantity only in the alkaline hydrolyzates of members of the *Salicaceae* family of the dicotyledonous angiosperms. The present finding of *p*-hydroxybenzoic acid in the hydrolyzate of the monocotyledonous angiosperm, *Sabal palmetto*, led us into a study of the monocotyledons in general and the palms and bamboos in particular. The present study reports results on the alkaline hydrolysis of representative members of the *Palmaceae* family.

The high yield of *p*-hydroxybenzoic acid in the alkaline hydrolyzate of trunk material of *Sabal palmetto* indicated this abundant species as a possible commercial source of this acid. It also led us to speculate concerning the occurrence of *p*-hydroxybenzoic acid in other palm species and to determine whether the acid was specific to *Sabal palmetto* or generic to all palms. A search for trunk material of other palm species indicated that the tree-sacrifice required for the obtainment of such material would make such obtainment almost impossible in the cases of the less common species. However, it became apparent that the woody leaf stalks or petioles of many palms were available, and these were suggested as starting materials for preliminary studies on palmwood chemistry. Accordingly, *Sabal palmetto* petiole material was hydrolyzed with boiling normal sodium hydroxide, and the hydrolyzate analyzed.

IRWIN A. PEARL, Senior Research Associate and Chief; DONALD L. BEYER, Research Aide; and DAWN LASKOWSKI, Laboratory Assistant, Lignin Chemistry Group, The Institute of Paper Chemistry, Appleton, Wis.

Table I. Alkaline Hydrolysis of *Sabal palmetto*

	Yield,[a] %	
	Trunk material	*Petiole material*
Total ether extract	3.8	3.3
Vanillin	0.9	0.4
Syringaldehyde	1.3	1.5
p-Hydroxybenzaldehyde	0.5	0.3
Acetovanillone	0.3	0.3
Acetosyringone	0.5	1.1
Vanillic acid	1.3	1.3
Syringic acid	3.1	1.3
Ferulic acid	2.2	0.0
p-Hydroxybenzoic acid	81.3	11.5

[a] The yield of total ether extract is on the basis of oven-dry starting material. All other yields are on the basis of the total ether extract.

Qualitative chromatography indicated essentially the same phenolic materials in the hydrolyzate as were found in the hydrolyzate of the trunk material. Only ferulic acid was missing in the petiole hydrolyzate. In the case of the petiole hydrolyzate, however, quantitative chromatography indicated that *p*-hydroxybenzoic acid did not comprise the very high percentage of the total ether extract produced on hydrolysis as it did in the case of the trunk material. Nevertheless, it was still the chief component of the ether extract. Comparison data are found in Table I.

EXPERIMENTAL PROCEDURE

Petioles from 26 representative palm species were obtained from Fairchild Tropical Garden in Miami, Fla., and the USDA Plant Introduction Stations in Miami, Fla., and Savannah, Ga. Finely divided petiole material was subjected to alkaline hydrolysis as described earlier (*1*). Hydrolyzates were acidified and extracted with ether. Ether extracts were chromatographed

Table II. Chromatographic Data for Hydrolyzate Extractives in Butanol-Aqueous Ammonia

	Butanol-2% aqueous ammonia R_f[a]									
	0.00	0.08[b]	0.10[c]	0.12[d]	0.14[e]	0.38[f]	0.44[g]	0.60[h]	0.76	0.88
Acrocomia armentalis	X	X	X	X	X	X	X	X	..	X
Arecastrum romanzoffianum	X	X	X	X	..	X	X	X	..	X
Arenga pinnata	X	X	X	X	..	X	X	X	..	X
Butia bonneti	X	X	X	X	..	X	X	X	X	X
Butia capitata[i]	X	X	X	X	X	X	X	X	X	..
Cocos coronata[i]	X	X	X	X	X	X	X	X	..	X
Cocos nucifera	X	X	X	X	X	X	X	X	X	X
Corypha talliera	X	X	X	X	..	X	X	X	..	X
Elaeis guineensis	X	X	X	X	X	X	X	X	..	X
Hyophorbe amaricaulis	X	X	X	X	X	X	X	X	..	X
Inodes causiarum[j]	X	X	X	X	X	X	X	X	..	X
Latania commersonii	X	X	X	X	X	X	X	X	..	X
Linoma alba, var. *aurea*[k]	X	X	X	X	X	X	X	X	..	X
Livistona saribus[k]	X	X	X	X	X	X	X	X	..	X
Nanhoraps richardiana	X	X	X	X	X	X	X	X	..	X
Nannorhops ritchiana	X	X	X	X	X	X	X	X	..	X
Opsiandra maya	X	X	X	X	X	X	X	X	X	..
Phoenix dactylifera	X	X	X	X	X	X	X	X	..	X
Phoenix pusilla[i]	X	X	X	X	X	X	X	X	X	X
Pseudophoenix sargenti	X	X	X	X	X	X	X	X	..	X
Pseudophoenix saonae	X	X	X	X	X	X	X	X	..	X
Ptychosperma sp.	X	X	X	X	X	X	X	X	X	..
Rhapidophyllum hystrix	X	X	X	X	X	X	X	X	X	X
Roystonea regia	X	X	X	X	X	X	X	X	..	X
Sabal palmetto[i]	X	X	X	X	..	X	X	X	X	..
Serenoa repens	X	X	X	X	..	X	X	X	X	X

[a] R_f determined by means of bis-diazotized benzidine spray.
[b] Syringic acid.
[c] Vanillic acid.
[d] *p*-Hydroxybenzoic acid and ferulic acid.
[e] *p*-Coumaric acid.
[f] Syringaldehyde and acetosyringone.
[g] Vanillin.
[h] Acetovanillone.
[i] This hydrolyzate also gave a spot at R_f 0.27.
[j] This hydrolyzate yielded a bright purple spot with bis-diazotized benzidine and with diazotized *p*-nitroaniline sprays at R_f 0.80.
[k] This hydrolyzate also gave a spot at R_f 0.82.
[l] This spot did not contain ferulic acid.

qualitatively on paper and developed in butanol saturated with 2% aqueous ammonia and in 10:3:3 butanol-pyridine-water systems. Spots were located by means of bis-diazotized benzidine, diazotized *p*-nitroaniline, 2,4-dinitrophenylhydrazine, and Mäule spray reagents and by examination under ultraviolet light. Ether extracts were also qualitatively chromatographed for *p*-hydroxybenzaldehyde in a *n*-heptane-*n*-butyl ether-water developer as described earlier (*2*). Qualitative chromatographic data are given in Tables II and III. Where found, vanillin, syringaldehyde, *p*-hydroxybenzaldehyde, acetovanillone, acetosyringone, vanillic

Table III. Chromatographic Data for Hydrolyzate Extractives in 10:3:3 Butanol-Pyridine-Water

	10:3:3 Butanol-pyridine-water R_f[a]									
	0.00	0.04	0.09	0.18	0.27	0.38[b]	0.44[c]	0.56[d]	0.78[e]	0.84[f]
Acrocomia armentalis	X	..	X	X	X	X	X	X	X	X
Arecastrum romanzoffianum	X	..	X	X	X	X	X	X	X	X
Arenga pinnata	X	..	X	..	..	X	X	X	X	X
Butia bonneti	X	X	X	X	..	X	X[g]	X	X	X
Butia capitata	X	X	X	X	X	X	X	X	X	X
Cocos coronata	X	..	X	X	..	X	X	X	X	X
Cocos nucifera	X	..	X	X	X	X	X	X	X	X
Corypha talliera	X	X	X	X	..	X	X	X	X	X
Elaeis guineensis	X	X	..	X	..	X	X	X	X	X
Hyophorbe amaricaulis	X	X	X	X	X	X	X	X	X	X
Inodes causiarum[h,i]	X	..	X	X	X	X	X	X	X	X
Latania commersonii[i]	X	X	X	X	..	X	X	X	X	X
Linoma alba, var. *aurea*[i]	X	X	X	X	..	X	X	X	X	X
Livistona saribus	X	X	X	X	..	X	X	X	X	X
Nanhoraps richardiana	X	..	X	X	..	X	X	X	X	X
Nannorhops ritchiana	X	..	X	X	..	X	X	X	X	X
Opsiandra maya	X	X	X	X	X	X	X	X	X	X
Phoenix dactylifera	X	..	..	X	..	X	X	X	X	X
Phoenix pusilla	X	X	X	X	X	X	X	X	X	X
Pseudophoenix sargenti[i]	X	..	X	X	..	X	X	X	X	X
Pseudophoenix saonae	X	..	X	X	..	X	X	X	X	X
Ptychosperma sp.	X	X	..	X	X	X	X	X	X	X
Rhapidophyllum hystrix	X	X	X	X	..	X	X	X	X	X
Roystonea regia	X	X	X	X	..	X	X	X	X	X
Sabal palmetto	X	X	X	X	X	X	X[g]	X	X	X
Serenoa repens	X	X	X	X	..	X	X	X	X	X

[a] R_f determined by means of bis-diazotized benzidine spray.
[b] Syringic acid.
[c] Vanillic acid and ferulic acid.
[d] *p*-Hydroxybenzoic acid and *p*-coumaric acid.
[e] Syringaldehyde, acetosyringone, and other compounds.
[f] Vanillin, acetovanillone, and other compounds.
[g] This spot did not contain ferulic acid.
[h] This hydrolyzate gave a bright purple spot with bis-diazotized benzidine and with diazotized *p*-nitroaniline at R_f 0.74.
[i] This hydrolyzate gave a spot at R_f 0.12.

Table IV. Alkaline Hydrolysis of Petioles of Representative Palms

Scientific name	*Common name*	*Source*[a]	*Ether extract, %*[b]	*Vanillin, %*[c]	*Syringaldehyde, %*	*p-Hydroxybenzaldehyde, %*	*Acetovanillone, %*	*Acetosyringone, %*	*Vanillic acid, %*	*Syringic acid, %*	*p-Hydroxybenzoic acid, %*	*Ferulic acid, %*	*p-Coumaric acid, %*
Acrocomia armentalis	Cuban acrocomia	II	2.4	0.7	0.7	0.5	0.3	1.1	2.2	3.6	25.8	2.0	1.7
Arecastrum romanzoffianum	Queen palm	II	1.6	1.0	1.6	0.7	0.6	1.2	3.1	3.2	39.0	4.5	1.6
Arenga pinnata	Sugar palm	III	1.7	1.1	1.0	0.5	0.7	1.4	2.6	2.6	55.4	3.1	0.0
Butia bonneti	...	III	3.2	0.2	0.3	0.4	0.5	0.5	1.3	2.4	72.0	1.6	0.0
Butia capitata	Butia palm	I	2.6	0.6	1.0	0.0	0.5	0.9	1.8	3.2	46.7	1.6	1.3
Cocos coronata	...	II	2.9	0.3	0.3	0.0	0.8	0.5	3.3	3.3	20.8	2.5	1.5
Cocos nucifera	Coconut palm	II	2.8	0.3	0.4	0.0	0.3	0.6	2.4	2.8	25.4	2.8	2.6
Corypha taliera	...	II	2.5	0.2	0.4	0.0	0.5	0.4	2.6	2.8	37.9	2.5	0.0
Elaeis guineensis	African oil palm	II	3.9	0.4	0.8	0.4	0.3	0.8	1.7	2.5	56.3	2.5	0.7
Hyophorbe amaricaulis	Bottle palm	II	3.6	0.6	0.6	0.5	0.4	0.3	3.2	1.7	31.7	2.6	0.5
Inodes causiarum	Puerto Rico Hat palm	II	3.6	0.5	0.5	1.0	0.5	1.6	1.4	2.4	63.4	1.7	0.6
Latania commersonii	...	II	3.3	0.3	0.3	0.6	0.2	0.7	2.4	2.6	44.5	2.9	1.7
Linona alba, var. *aurea*	...	II	4.0	0.5	0.2	0.8	0.3	0.4	1.9	1.1	43.6	2.8	0.9
Livistona saribus	Fan palm	II	5.0	0.3	0.2	0.0	0.2	0.4	1.8	1.8	63.4	1.5	0.0
Nanhoraps richardiana	...	III	2.9	0.6	0.3	0.4	0.3	0.8	1.2	1.5	36.6	1.2	6.0
Nannorhops ritchiana	...	II	3.5	0.5	0.3	0.0	0.1	0.4	1.4	1.9	34.1	2.6	3.5
Opsiandra maya	...	II	2.8	0.2	0.2	0.0	0.3	0.2	1.8	1.0	13.1	1.8	0.6
Phoenix dactylifera	Date palm	II	1.9	0.3	0.6	0.0	0.5	0.9	2.4	2.3	43.4	2.7	1.0
Phoenix pusilla	Ceylon date palm	II	2.9	0.6	0.4	0.0	0.3	0.7	2.5	2.4	42.3	2.5	2.2
Pseudophoenix sargenti	Sargent cherry palm	II	2.3	0.4	0.4	0.8	0.4	0.7	1.7	2.2	32.3	3.4	0.6
Pseudophoenix saonae	...	II	2.5	0.2	0.2	0.0	0.6	0.6	2.1	1.9	23.8	3.5	1.3
Ptychosperma sp.	...	II	2.7	0.5	0.8	0.6	0.5	0.4	1.9	1.7	44.0	2.1	2.0
Rhapidophyllum hystrix	Needle palm	I	4.3	0.7	1.2	0.0	0.7	1.1	1.9	1.7	44.8	2.3	1.0
Roystonea regia	Cuban royal palm	II	2.2	0.6	0.4	0.0	0.5	0.9	2.3	2.7	40.5	2.9	1.7
Sabal palmetto	Cabbage palmetto	I	3.3	0.4	1.5	0.3	0.3	1.1	1.3	1.3	11.5	0.0	0.0
Serenoa repens	Saw palmetto	III	3.8	0.3	0.8	0.6	0.6	0.9	1.5	1.0	61.8	1.9	0.0

[a] Sources of palm petioles: (I) USDA,Barbour Lathrop Plant Introduction Station in Savannah, Ga.; (II) USDA, Plant Introduction Station in Miami, Fla.; (III) Fairchild Tropical Garden in Miami, Fla.
[b] The yield of ether extract is on the basis of oven-dry petiole material.
[c] The yields of vanillin and all other hydrolysis products are on the basis of the ether extract.

acid, syringic acid, *p*-hydroxybenzoic acid, ferulic acid, and *p*-coumaric acid were determined quantitatively by paper chromatographic and spectrophotometric procedures described previously (*1*, *2*, *3*). Quantitative data for these compounds are given in Table IV along with yields of total ether extracts obtained upon alkaline hydrolysis of the petioles.

DISCUSSION OF RESULTS

The yields of ether extractives obtained after alkaline hydrolysis of the petioles of the 26 representative palm species were somewhat higher than yields obtained from representative hardwoods under the same conditions (*1*, *2*). Yields ranged from about 2% to approximately 5% with an average yield around 3%.

Qualitative chromatography of the ether extractives demonstrated that, upon alkaline hydrolysis, all species examined yielded vanillin, syringaldehyde, acetovanillone, acetosyringone, vanillic acid, syringic acid, and *p*-hydroxybenzoic acid. In addition, every species yielded a compound or compounds still unidentified with R_f 0.00 in both the butanol-aqueous ammonia and butanol-pyridine-water developers. All palm petiole tested except that of *Sabal palmetto* yielded ferulic acid upon hydrolysis. This finding was unexpected in view of the fact that ferulic acid was produced by alkaline hydrolysis of trunk material of *Sabal palmetto* (see Table I). Para-hydroxybenzaldehyde, *p*-coumaric acid and several unidentified compounds noted in Tables II and III were found in only a portion of the species examined. A few compounds were very specific to a small number of species (see Tables II and III), and may have taxonomic significance. The species examined in this study have been tabulated alphabetically, and no attempt has been made to classify the individual species of genera botanically. Therefore, any apparent similarity of data for adjacent species or genera in the tables is coincidental.

Quantitative analysis data of Table IV are enlightening. In all cases *p*-hydroxybenzoic acid is the major component of the ether extract produced upon alkaline hydrolysis. In some instances the *p*-hydroxybenzoic acid comprises a very high percentage of the ether extract. It is interesting to note that the lowest yield obtained was from the hydrolysis of *Sabal palmetto* petioles—the same species whose trunk material yielded an ether extract containing more than 80% *p*-hydroxybenzoic acid. Thus, it appears that the trunk material of the other species tested might yield even higher percentages of *p*-hydroxybenzoic acid upon alkaline hydrolysis. Studies along this line are planned for the future.

The yields of all other compounds were relatively low. Aldehydes and acetophenones

were generally less than 1% of the ether extractives. Acid yields were somewhat higher, averaging around 2% of the extractives. In the case of *Nanhoraps richardiana* a yield of 6.0% *p*-coumaric acid was obtained from the ether extractives produced on alkaline hydrolysis. This yield was very unusual. The low yields for all compounds except *p*-hydroxybenzoic acid noted in Table I for *Sabal palmetto* trunk material indicate that trunk material of these 26 representative palm species would probably yield these compounds in approximately the same yields.

Syringyl-guaiacyl ratios demonstrated no regular pattern with respect to aldehydes, acetophenones, and acids for particular species. Similarly, the over-all picture for the 26 species is also indefinite. Syringyl-guaiacyl ratios are irregular in the cases of the aldehydes and acids, but are generally greater than one in the cases of the acetophenoles although there are several exceptions.

As noted earlier in connection with studies on the alkaline hydrolysis of representative hardwoods and their extractives (*4*) there apparently occur in all deciduous woods lignin and extraneous materials with guaiacyl and syringyl moieties containing side chain configurations capable of yielding both aldehyde and carboxylic acid products on hydrolysis with alkali alone without the presence of oxidizing agents. This also applies to the monocotyledenous palms, and would account for the yields of vanillin, syringaldehyde, vanillic acid, and syringic acid in the present study. In addition, the lignin of the monocotyledons (*5*) and probably the extraneous materials contain *p*-hydroxyphenyl moieties, and similar side chains on these structures would account for the *p*-hydroxybenzaldehyde and a little of the *p*-hydroxybenzoic acid found in the palm petiole hydrolyzates. The large yield of *p*-hydroxybenzoic acid found in these palm petiole hydrolyzates could not be due entirely to such a mechanism. Most of it must be a product of ester linkage hydrolysis as suggested by Smith in the case of *Populus tremula* hydrolysis (*6*).

It should be noted that the results reported herein, as in the representative hardwood studies, are preliminary in nature because, in all instances, they reflect experimental data from single specimens of individual species. Possible variations discussed earlier for hardwoods (*1*) apply to the results and discussions of this paper.

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Acidification of Paper Machine Shower Water with Sulphamic Acid to Prevent Blockage and Increase Felt Life

KENNETH A. CRAIG

A method of treatment of the felt shower water on a wide, high-speed book-paper machine is described which increased the felt life by two to threefold. Fill-up of felts on a paper machine may be caused by many circumstances. Each situation must be carefully surveyed and analyzed before corrective action can be taken. In the case of the no. 3 paper machine at Niagara, Wis., the felts were becoming filled with hydrous aluminum oxide. Laboratory tests predicted that the alkalinity in the untreated fresh water being used on the felt shower would react with the excess aluminum ions in the paper mill whitewater to form a hydrous aluminum floc *in situ* in the felts. Acidification of the fresh water used in the felt showers with sulphamic acid to pH = 5.2 resulted in a tremendous reduction in felt filling and increased the felt life by two- to threefold. Seldom has the author experienced such a spectacular improvement in mill operation brought about by a single change in operating conditions.

Production problems caused by operating fourdrinier paper machines under conditions causing rapid felt fill-up are universally recognized by the paper industry, as are resultant excessive costs of changing felts before they have worn out mechanically. Conditions which cause felt fill-up are less clearly understood.

Kenneth A. Craig, Chief, Paper and Newsprint Department, Research and Development, Kimberly-Clark Corp., Neenah, Wis.

Most such build-ups consist of pitch, fines, filler, or chemical additives in the furnish, either individually or in combinations that can vary from mill to mill. The latter may include common additives such as alum, clays, and chalk, or the newer wet-strength resins which may present more specialized problems. Each situation requires individual analysis.

This paper is presented to demonstrate how a typical felt filling problem was solved. By thorough analysis and technical studies, we were able to prescribe a successful solution by controlling the pH of felt shower water through continuous acidification with sulphamic acid. Annual savings on a single machine are well in excess of $100,000.

BACKGROUND

In August, 1955, we replaced the dual press section of our Quinnesec (Niagara, Wis.) mill's 226-in. no. 3 paper machine with a suction pickup roll, and a Twinver press. With this new transfer section and other

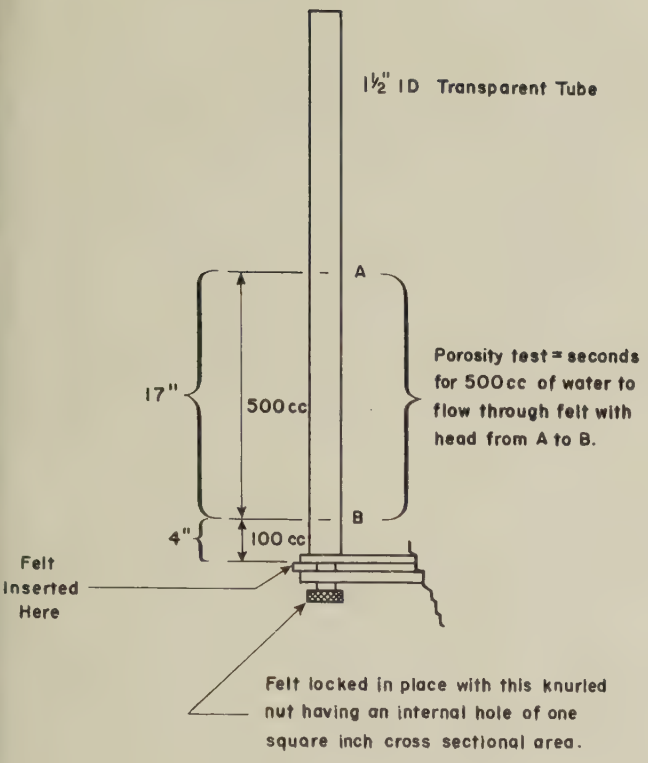
1½" ID Transparent Tube
A
17"
500 cc
Porosity test = seconds for 500 cc of water to flow through felt with head from A to B.
B
4"
100 cc
Felt Inserted Here
Felt locked in place with this knurled nut having an internal hole of one square inch cross sectional area.

Fig. 1. Porosity test

modifications, speeds above 1500 f.p.m. were achieved. Furnish consisted of 55% groundwood, 25% kraft, and 20% sulphite, for producing 43-lb., coated publication paper. Also included was the usual amount of coated broke.

Even before the change-over, clogging had kept felt life at an unacceptable level. Repeated washings with acids, alkalies, and detergents had not proved to be effective in removing the deposits. Working the felt by roping and turning had only been partially successful. Following the press replacement, the felt blocking or filling problem was greatly increased and soon became critical.

After being run for less than a week, felts either became hard and horny or filled up in spots causing crush or felt marks, requiring replacement. Essentially, all resiliency was lost. Based on a standard test (*see* drawing of porosity test) porosity was greatly reduced. A given volume of water took as much as 280 times longer to pass through a used felt as a new one (Fig. 1). As felts blocked, the suction pickup roll transfer failed to function properly. The sheet would fall away from the pickup felt, and breaks were frequent. Rough, uneven sheets and holes lowered paper quality. Poor drainage and water removal caused wet streaks resulting in uneven drying. This affected coating applications and necessitated production cutbacks to relieve overloaded driers. Uneven moisture distribution resulted in over drying, causing brittle spots which did not respond to smoothing in the breaker stack ahead of the two-side coater on this Quinnesec no. 3 machine.

Because of the pickup press roll arrangement, working or flexing could no longer be accomplished through roping or turning the felt. Periodic washing of the flat felt with chemicals resulted in production loss, and again proved relatively ineffective in cleaning or removing the "filler." Felt changes usually occurred within three to seven days, and pickup felts alone cost over $3000 apiece. At such short intervals, synchronizing felt changes with wire changes (approximately 9 days) was impossible. In addition to the three hours needed for a felt change, production loss also resulted from breaks when the sheet did not adhere to the pickup felt and from reduction in machine speeds to allow proper water removal.

There were several obvious reasons for the sudden intensification of the fill-up problem. A vacuum pickup is more likely to cause filling than a dual or a straight through press since it pulls a tremendous amount of water through the felt, simultaneously sucking in fines and other particulate filterable materials. Pitch is concentrated in the fines. When the paper sheet is formed, the coarser fibers collect against the wire, and the fine fibers filter to the top. On a dual or straight through press, the felt contacts the same side of the sheet as the wire (coarse fibers). In this Quinnesec no. 3 vacuum pickup, the sheet is sucked from the wires to the felt so that the fines side of the sheet contacts the felt. This impingement and the high water removal at the pickup felt press roll is then free to cary more materials into the felt (*see* Fig. 2).

EXPERIMENTAL DETAILS

Quite a bit of reaearch time had been spent in an effort to solve the felt problem. The usual static tests of trying to clean swatches of filled-up felts with chemicals in beakers confirmed plant experience. A dynamic testing system more typical of actual mill conditions was necessary.

A laboratory felt washing unit was built consisting of two mechanically driven squeeze rolls and a tension mounted about four feet apart. A pan was provided so felts could be saturated with paper mill white water. A spray shower for treating the felt with various washing materials and a small scale Uhle box for removing water were also provided. A 10-in. by 10-ft. belt of felt was run at 86 f.p.m.

Again, the commonly recognized chemical cleaners showed little effectiveness in removing contaminates from felts removed from the Quinnesec no. 3 paper machine. It was concluded that a new approach had

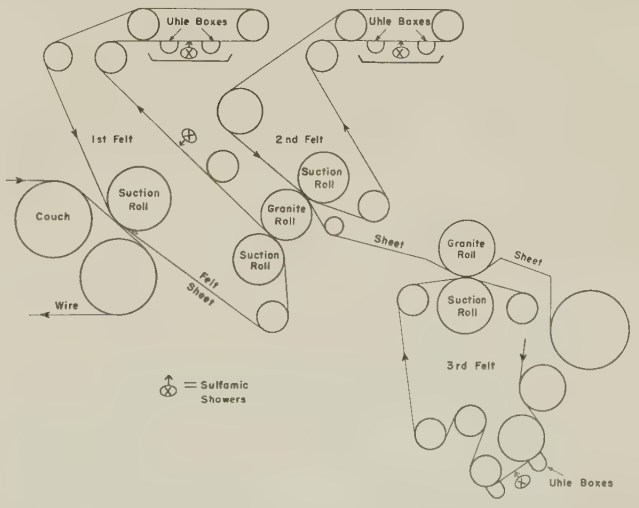

Fig. 2. Kimberly-Clark Niagara mill—Q-3 machine sketch of wet press section

to be made. The contaminates had to be prevented from building up in the felt.

This necessitated an analytical study of foreign materials deposited on the felt fibers. At first it was suspected that pitch was responsible. Solvent extractions of the dried felts proved that even the most severly plugged felts had low pitch contents (0.2 to 0.7%).

Further analysis of several felts showed a very high ash content of 12 to 18% based on the total dry weight. Alum deposits were suspected, and an analysis of the white water was made which showed 60 p.p.m. of soluble aluminum ions. This content resulted from a high alum addition rate which had been found to be optimum and consistent with best operating experience in the mill to prevent picking at the granite press rolls. To establish that the ash fouling the felt was coming from the added alum rather than the base clay and paper coating clay entering the system from the broke, additional analyses were necessary. By determining the ratio of SiO_2:Al_2O_3 in the paper sheet, and the same ratio in the felt deposit, the excess Al_2O_3 could be assumed to have been deposited from precipitated aluminum ions. These analyses proved that in this situation, over 80% of the Al_2O_3 in the felt was being precipitated from the alum in the white water.

With this information, it was possible to form the hypothesis that the soluble aluminum ions (simple Al^{+++} or hydroxylated $Al(OH)_x$) react with the alkalinity in the fresh mill water being used in the felt showers to form a gelatinous $Al(OH)_3 \cdot xH_2O$. This may be illustrated by the following equation:

$$Al^{+++} + O\overline{H} \rightarrow Al(OH)_3 \cdot xH_2O \downarrow \quad \text{or} \quad Al(OH)_x$$

The alkalinity may come from hydroxyl ions (free or hydrous) or from carbonate and bicarbonate ions which are present in untreated mill process fresh water at pH = 8.0. The word alkalinity is used even though the fresh water might be at a pH below 7.0 since alkalinity in water chemistry is defined as the titratable quantity to the methyl orange end point (pH = 4.3).

This gelatinous precipitate is formed *in situ* on the felt fibers. As it remains on the surface, this inorganic residue not only collects other filterable material but undergoes further polymerization while splitting off water. As this process progresses, the deposit becomes increasingly difficult to dissolve, which explains the relative ineffectiveness of periodic washings.

Based on this reasoning, it was decided that adjusting the shower wash water pH to that of the white water would keep basic aluminum ions dispersed and in a readily soluble form that could be continuously washed from the felt. The pH adjustment would neutralize the hydroxyl ions and dispel the carbonate ions as CO_2 gas. This would be a preventative treatment, as opposed to the actual removal of plugging materials once they have formed.

Two relatively simple laboratory tests were conducted to affirm the above hypothesis. First, paper mill white water was filtered, and unneutralized fresh mill water with an alkalinity equal to that found in the mill showers (pH 8.0) was added. As suspected, a gelatinous precipitate formed. When the shower water was acidified to a pH of 5.0 with sulphamic acid, the mixture remained clear. This substantiated our hypothesis.

Further proof was obtained with the laboratory felt washing machine. We started with a clean felt, running it through paper mill white water containing soluble aluminum ions in the saturation pan. Again, fresh water, with a pH of 8.0, was added, this time through the shower. A deposit quickly built up in the felt. The felt had a hard, horny hand, and there was a noticeable drop in porosity values. The ash showed high quantities of Al_2O_3. A second run was made on a new felt with the shower water adjusted to a pH of 5.0. This second test produced no build-up.

We were now ready to make a mill trial. The remaining question concerned which acid to use.

It is well known that sulphates will precipitate basic aluminum ions, which could also cause problems. Based on analogous studies in the literature with nitrates and chlorides, we knew the basic sulphamate salts should be more soluble. It was further known that the sulphates of calcium (present from the calcium carbonate containing broke) were considerably less soluble than the sulphamates. This pointed toward sulphamic acid.* Also, during our attempts to chemically remove the build-up in the felts, we had noted that sulphamic was one of the best felt-washing materials available, and that it seemed to give felts a softer hand. We therefore decided to suggest its use. Although it is more expensive than other acids, its advantages outweigh this consideration.

We recommended that a sulphamic acid solution be introduced into the felt shower water in sufficient quantities to lower the pH to 5.2 so the precipitate could not form. The paper mill personnel were also in favor of using sulphamic because of its freedom from the dangers of fumes and liquid spillage. No special dilution equipment is necessary, and it is somewhat less corrosive than other acids. Available in non-returnable fiber drums, it is safe and easy to store and handle.

There had been two previous attempts made on Quinnesec no. 3 machine at such pH control with sulphamic, but neither was carried out under optimum conditions. In one case a relatively strong solution was introduced for approximately two hours each day. This represented an attempt to clean a filled-up felt. During the second trial, a relatively weak solution had been sprayed onto a filled felt with more continuity, but only in sufficient quantities to lower the pH to approximately 6.0. This emphasizes the need for complete testing over an extended period of time and with adequate controls to assure consistent application.

EQUIPMENT

A make-up system consisting of a mix tank, strong acid storage tank, and weak acid storage tank was designed and installed. A batch of acid solution is prepared in the mix tank using 80 lb. of the granular acid to 160 gal. of water. During the mixing, steam is applied, elevating the water temperature to approx-

* Sulphamic acid is a white, nonhygroscopic, nonvolatile, solid, inorganic acid. It is available either in crystalline or granular form. When dissolved in water, it forms highly ionized solutions which approach the strength of hydrochloric, sulphuric, or nitric acids. Originally prepared more than 100 years ago, sulphamic acid was little more than a laboratory curiosity until 1940. It is produced by DuPont's Industrial and Biochemicals Dept.

imately 120°F. This assures immediate and complete dissolving of the granules.

Once mixed, the solution, with a pH of 0.7, is fed into the 400-gal. strong acid storage tank. From there it is pumped into the weak acid storage tank as needed.

The latter has a capacity of 900 gal., and is equipped with a high-low level control on the warm, fresh water inlet. The pH is kept at 2.2(1.6 lb. of acid per 100 gal. of water). A pH meter further along in the felt shower water line activates a variable speed metering pump that controls the final shower water at a pH of 5.2. All three felts on this Quinnesec no. 3 machine are treated. Approximately 250 g.p.m. are used on all three felt systems of this paper machine, with about

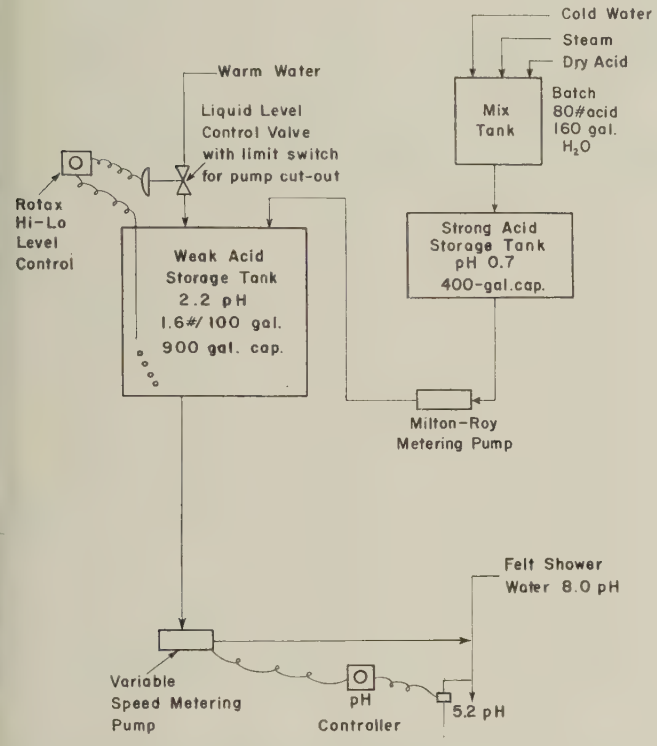
Cold Water
Steam
Dry Acid
Warm Water
Liquid Level Control Valve with limit switch for pump cut-out
Mix Tank
Batch 80#acid 160 gal. H_2O
Rotax Hi-Lo Level Control
Weak Acid Storage Tank 2.2 pH 1.6#/100 gal. 900 gal. cap.
Strong Acid Storage Tank pH 0.7 400-gal.cap.
Milton-Roy Metering Pump
Felt Shower Water 8.0 pH
Variable Speed Metering Pump
pH Controller
5.2 pH

Fig. 3. Flow diagram of sulphamic acid make-up system at Kimberly-Clark Niagara Mill—Q-3 machine

125 g.p.m. being used on the pickup felt. All shower water is heated to approximately the temperature of the white water. Equipment and piping handling the strong acid solution is made from no. 316 stainless steel. Piping handling the dilute solution in the main shower line is made from no. 304 stainless. (*see* Fig. 3.)

RESULTS

An immediate and marked improvement was noted following our change-over to all treated shower water on the pickup felts on Feb. 9, 1956, as shown in Table I and Fig. 4. Since that time, felts have consistently lasted the life of two wires, and it is not uncommon for them to last for three. In many cases the felts could have run longer than indicated on the chart, but the determining factor on their removal was the need for a wire change or other mechanical failures.

It is seldom that a change as abrupt as this is observed in mill operations, and even less often that we have the

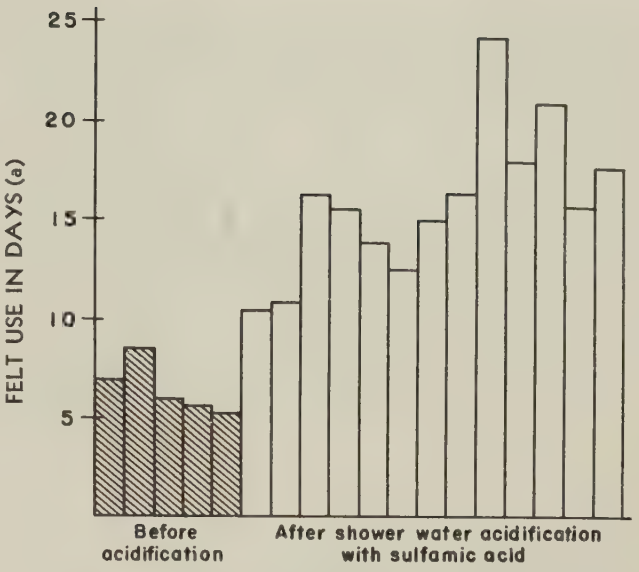

Fig. 4. Kimberly-Clark—Q-3 paper machine felt use record

(a) Before acidification, felt use time was usually limited by the felt becoming too filled up for further operation. After acidification, maximum life is not always indicated because felts with life remaining were changed to coincide with wire changes or other shutdowns.

fundamental data to explain the observed change. The three to four fold reduction in both ash and washability and the almost logarithmic reduction in porosity as a function of ash are only part of the substantiating evidence. The general appearance and feel of the felts are also entirely different since the sulphamic acid treating was begun.

While corrosion of framework and concrete floors can be a problem, this can be handled by using resin coatings, corrosion-resistant paints, and asphalt based floor coverings.

The basic principles learned in solving this particular felt blocking problem have been applied to other situations in some of our other mills with successful results. It must be emphasized that each set of conditions must be studied and understood individually before a sound preventative treatment can be pre-

Table I. Untreated Shower Water

Days of felt use	*Pitch, %*	*Ash, %*	*Porosity[a] time, sec.*	*Washability,[b] %*
3	0.29	12.6	988	15.8
4	0.69	11.9	228	10.3
6	0.28	17.6	2709	17.1
7	0.31	16.3	2813	22.4
7	0.32	16.1	1153	16.3
4	0.29	14.1	490	15.5
5	0.19	14.6	773	19.4
5	0.37	13.4	454	20.9
5	0.42	15.2	853	19.6
5	0.42	12.9	272	18.3
		Sulphamic Treatment Begun		
8	0.56[c]	4.5	75	5.0
9	0.43[c]	3.8	56	4.2
10	1.03[c]	5.3	35	5.9
13	1.15[c]	3.1	67	4.3

[a] Porosity of a new felt ranges from 10 to 20 sec.
[b] The percentage material removed on washing in an agitator type of washing machine for 1 hr. Percentage based on O.D. total weight.
[c] Increased pitch pick-up is to be expected as felt life is extended.

scribed. In the case under discussion, the problem was caused by alum and the alkalinity of the fresh water; in another, it may be pitch or a wet-strength resin.

In many of these conditions, we are dealing with colloidal systems of alum or pitch, where alum is used to control pitch, as in newsprint and other groundwood systems. The importance of maintaining a proper pH of shower wash water must, therefore, always be kept in mind.

SUMMARY

The felt life on a high-speed and wide book—paper machine was very short due to filling with hydrous aluminum oxide. The introduction of sulphamic acid into the felt shower water increased the life by two to threefold.

RECEIVED July 6, 1959.

The Morphology of Hardwood Fibers

PAUL W. LANGE

The morphology of the woody cell with emphasis on the structure of fibers in hardwoods has been reviewed, and methods of determining the fibrous structure and its effect in paoermaking are discussed.

IT IS quite obvious that a thorough knowledge of the plant cell wall structure is required in order to fully understand the complicated processes which occur during chemical or other treatment of wood and it should be emphasized—even if it may seem self-evident—that, in general, wood chemical reactions of importance occur within the cell wall. It is only during the past 30 years that it has become evident to industry that this knowledge about the minute structure of wood is of the utmost technical importance. During this time extraordinary advances have been made in the knowledge of the architecture of the plant cell wall mainly as a consequence to the new tools, which modern physics has made available.

Morphological problems should therefore be of interest not only to specialized botanists but also to the practical man in the cellulose and paper industry. Without a certain knowledge about the morphology of the woody tissue and its components a deeper knowledge of many technical processes is not possible.

As an example it may be mentioned that a proper understanding of pitch problems in connection with the pulping of wood is not possible without a thorough knowledge of the localization of the resins in the wood in minute details (*1*).

The present paper deals with the morphology of the woody cell with emphasis on the cell wall structure and with special reference to the hardwoods.

Most work on wood fiber structure has been carried out with the softwood tracheid as the object. Since hardwood fibers in many respects are similar to softwood tracheids the structure of the former should be compared with that of the latter.

TYPES OF CELLS IN HARDWOODS AND SOFTWOODS

Hardwoods are characterized by greater complexity of structure than softwoods. The woody tissue in hardwood is a younger and more specialized type of tissue than that in softwoods. In the latter tissue the function of support and conduction is performed by one type of element, the thick walled and thin walled tracheid, respectively, which make up about 90% of the woody tissue.

Hardwoods have a characteristic element, the vessel, formed for conduction purposes. Vessels are broad tubes which can be several meters in length. They conduct water vertically up the tree trunk. Tracheids are also present in hardwoods and serve more as a means for conduction than support, a function associated with the thicker-walled fibers. Intermediate cells known as fiber-tracheids are also present and from a technological point of view the difference between tracheids, fiber-bracheids, and fibers can generally be ignored. They are often all denoted as fibers.

As in softwoods the storage tissue consists of horizontal (ray cells) and vertical parenckyma cells. They are much shorter than the fibers and are of ten much more abundant in hardwoods than in softwoods and also how a greater variety of form. Figure 1 shows various types of nonlining cells in hardwoods.

In many cases the fibers account for the bulk of the cells in hardwoods. Since from a technological point of view the fibers are the most important elements, the morphology of these elements exclusively will be discussed in this paper.

FORM AND STRUCTURE OF HARDWOOD FIBERS

Fibers are elongated cells. Their form and structure cannot be completely understood without taking into consideration the manner in which they have been formed during growth. In this paper, however, the factor of growth is only briefly mentioned and no detailed attention will be paid to its influence upon the fiber structure.

Hardwood fibers are shorter than softwood tracheids (0,5 to 2 mm, and 2–5 mm., respectively). The thickness of the cell wall in hardwood fibers varies considerably in different species (average values 1,5 to 10 μ). In general, the hardwood fibers can be compared with the summerwood tracheids with respect to the thickness of the cell wall.

It seems now to be a generally accepted fact that cellulose—more or less "pure"—is deposited in the dell wall in the form of microfibrils, 50 to 150 A thick and of indefinite length. From a chemical point of view they cannot be considered as basic units, since microfibrils from different cellulose origins can be somewhat different in chemical character. Thus microfibrils from wood

PAUL W. LANGE, The Central Laboratory, Aktieselskapet Borregaard, Sarpsborg, Norway.

Fig. 1. Various types of nonliving cells in hardwoods a, b vessels, c, d tracheids, e, f fibers

cellulose are different from those of cotton cellulose.

The cellulose microfibrils represent most of the highly organized ordered material in the fiber. Hemicelluloses and lignin seem to be deposited in a more disordered way. However, Meier (*2*) has shown that hemicelluloses can also be organized in microfibrils of the same dimensions as cellulose microfibrils. Thus a resistant fraction of mannan builds up the frame work of the cell walls of palm seeds in the form of microfibrils. Meier states that this might possibly be an indication that the resistant hemicelluloses of woody fibers also form microfibrils (Fig. 2).

The organization of the carbohydrates in the form of microfibrils or incrusting material in the cell wall and the deposition of other cell wall components, mainly lignin, seems to be one of the most important subjects for fiber morphology today. An outline of the present knowledge in this field is given in the following. Although this knowledge is a result of many different methods of investigation, e.g., x-ray and polarization optical methods, emphasis will be laid upon results ob-

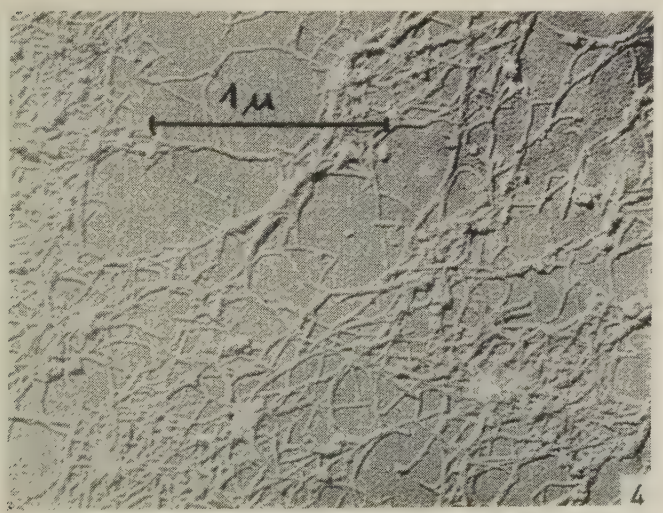

Fig. 2. Microfibrils of mannan B [Meier (2)]

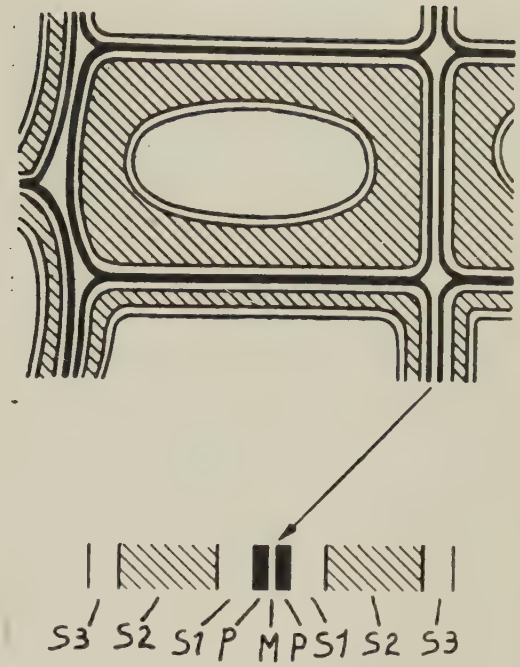

Fig. 3. Cross section of a fiber. Terminology according to Kerr and Bailey (12)

tained during the last decade with electron optical methods.

For a detailed knowledge the reader is referred to the literature (*3–11*).

THE CELL WALL LAYERS

Terminology

The structure of the plant cell wall is generally described with reference to different cell wall layers, considered to have a more or less individual character. The terminology generally used in this connection is that of Kerr and Bailey (*12*), who describe the cells in a woody tissue in terms of the middle lamella, which is the intercellular material, the primary wall, which envelops the secondary wall, which in turn is composed of three layers, the outer (S_1), middle (S_2), and inner secondary wall (S_3) (Fig. 3). The terminology of the three latter layers has been extensively discussed lately.

The Middle Lamella

The middle lamella is the name of the material between the fibers in a woody tissue. It is often difficult to distinguish between this "true middle lamella" and the "compound middle lamella" which also includes the two contiguous primary walls on both sides of the "true middle lamella." The thickness of the compound middle lamella varies between a few tenths of a micron and about two microns. During commercial pulping the middle lamella is more or less dissolved and the individual fibers are considerably loosened from each other.

According to Bailey's (*13*) microanalysis the middle lamella in wood of Douglas-fir contains 72% lignin and 14% pentosans. The rest was shown to be hemicellulose of glucoronic type. Lange (*14*) measured the lignin content in the native middle lamella of Swedish spruce ("compound middle lamella") and found values between 60 and 90%. The range of these values is not only a measure of the error of the microoptic measurements but certainly also shows the great biologic varia-

tion in the chemical composition of the compound middle lamella. Cellulose seems to be completely absent in the middle lamella.

The lignin seems to be isotropically deposited in the middle lamella. A certain orientation, however, cannot be excluded, since Lange (*15*) has measured a small but significant degree of intrinsic dichroism in ultraviolet light for native lignin in the middle lamella of Swedish spruce.

Ultrathin sections of enzymatically treated middle lamella of Swedish spruce impregnated with osmium were investigated by Asunmaa (*16*). The distribution of metal deposited in the middle lamella was random and no sign of any orientation could be discovered. The most frequent distance between the metal particles was 60 to 70 A. The structure must therefore be considered as amorphous. It should be observed, however, that the fine structure shows the structure of the material in the middle lamellas left after the enzymatic treatment.

Most of the investigations concerning the structure of the middle lamella have been made on softwoods. There are no reasons, however, to believe that great differences in the general architecture of the middle lamella in hardwoods compared to the softwoods should exist.

The Primary Wall

The primary wall is the outermost layer of the isolated individual cell. There is a fundamental difference between the primary wall and the secondary wall with respect to the formation and growth. The primary wall grows in area and very little in thickness while the secondary wall grows in thickness and not in area. This very important difference in growth may have some influence upon the properties of the microfibrils in the two layers.

In the dry state as it is seen in the electron microscope the primary wall is only about 300 A thick, i.e., the thickness of three microfibrils.

In the native state, however, the micro fibrils in the primary wall are highly individualized and separated from each other. The thickness of the native primary wall is estimated to about 0.1 μ.

The cellulose content of the primary wall in wood fibers is certainly below 50%. The hemicelluloses consist of mostly mannan and xylan in spruce and birch, respectively. The primary wall is further heavily lignified.

From polarization optical measurements it has been assumed that the microfibrils are transversally oriented in the primary wall. Preston (*3*) showed that there is a great angular dispersion in the organization of the fibrils, i.e., they cannot be parallelly arranged with respect to each other. The microfibril structure must therefore be described as a loose network. Further, electron optical investigations have shown that the network in the outer layers of the primary wall has a tendency to a direction parallel to the fiber axes while the microfibrils deposited in the layers toward the secondary wall are more transversally organized.

It is well known that the primary wall swells very little and that it changes its dimensions only to a small degree during delignification. Even Cuoxam dissolves the primary wall slowly. This resistance of the primary wall in swelling media is—at least partly—due to its network structure.

Meier (*6*) showed that the microfibrils in the primary wall of spruce and birch were more resistant toward enzymatic degradation than those in the secondary wall and he concluded that the primary wall cellulose is not identical with that of the secondary wall.

This difference in resistance is not fully explained. One possibility is that the chemical composition of the two kinds of microfibrils is different, the microfibrils of the primary wall also containing hemicellulose units. If both kinds of fibrils contain only cellulose chains as building material, the difference in chemical resistance against fungal attack may be explained as a result of the different ways in which the two walls are formed. It is highly probable that the fibrils in the primary wall have grown under a certain action of tension. It cannot be excluded that part of the fibrils in the primary wall consist of hemicellulose chains, as was mentioned above (Meier (*2*)).

The Secondary Wall

It is a generally accepted fact that the wall enclosed by the primary wall generally consists of three layers. In a transverse section of a tracheid or a fiber these three layers show different optical properties. According to the terminology of Kerr and Bailey these layers are referred to as the outer (S_1), middle (S_2), and inner (S_3) layers of the secondary wall. This terminology has been accepted by most workers on cell wall structure. However, for instance, Bucher (*17*) and Meier (*6*) do not consider S_1 and S_3 as parts of the secondary wall but as morphological entities fundamentally differing from S_2. They refer to S_1 and S_3 as transition lamella and tertiary wall, respectively.

As was said above the secondary wall grows in thickness and not in area. The thickness varies roughly between 1 and 10 μ. The microfibrils in this layer are highly parallel and more closely packed than in the primary wall. The content of cellulose in the secondary wall is considerably higher than that in the primary wall. The secondary wall of hardwoods is less lignified than that of softwoods but seems to have a slightly higher content of hemicelluloses.

The main direction of the microfibrils in S_1 (outer layer of the secondary wall or transition lamella) was previously thought to be along a flat helix. There is, however, new evidence of a crossed fibrillar structure, i.e., one Z-helix and one S-helix, both more or less flat, for instance in birch (*6*), but also in pine (*8*). It is not known how widespread this crossed fibrillar structure is. From x-ray measurements Preston (*3*) stated that there is a great angular dispersion with respect to the direction of the microfibrils in S_1.

Wardrop and Dadswell (*9*) pointed out that the number of microfibrils in the two directions of S_1 must differ considerably, which was also found by Meier (*6*) in the case of birch fibers. This latter statement seems to be supported by the fact that only one direction of S_1-helices has been observed in balloon swelling of wood fibers.

Much remains to be done in connection with this problem on the crossed fibrillar structure. According to Wardrop and Dadswell (*9*) there seem to be considerable technical difficulties associated with the determination of the organization of S_1, both with respect to the preparation of suitable specimens and with respect to the

interpretation of the results obtained. For instance, in sectioned material it is difficult to see if $S_1 + S_2$ or only S_1 is examined. In the former case the effect of a crossed fibrillar structure would be obtained.

The S_1-layer is like the primary wall, strongly lignified. Meier (*6*) showed that the microfibrils of S_1 are more resistant against fungal attack than those of S_2. The reason may be a difference in the physicochemical structure of the microfibrils and/or the incrusting material. It is further remarkable that S_1 is more resistant than S_2 in swelling media (balloon swelling). In this case it should be observed, however, that the stress, which S_1 is subjected to during the swelling, may play an important role.

The dominating part of the cell wall is the layer S_2. The greatest part of the cellulose microfibrils is deposited in S_2 in a closely packed highly parallel screw structure for birch. The angle between the fiber axis and the direction of the microfibrils is only a few degrees. Preston (*3*) reported the value of 18° for this angle in the S_2-layer of spruce tracheids.

The S_2-layer in hardwoods is generally less lignified than that of softwoods. This difference in lignification is, for instance, associated with a difference in the decay of wood by fungus (*6*). On the other hand there seem to be more hemicelluloses deposited in S_2 in the case of hardwoods.

It has frequently been observed that S_2 consists of concentric lamellas of cellulose. This lamellation, which is easy to observe in a swollen cell wall, is considered to be the result of a small differentiation in fibril direction.

According to Wardrop and Dadswell (*9*) lamellas of only one or two sheets of microfibrils in S_2 of certain hardwoods have been observed as have also bigger aggregates of fibrils, which probably are the origin of striations observed in the light microscope. This lamellation does not seem to be connected with day and night deposition of material in the S_2 layer.

The innermost layer of the cell wall (S_3) sometimes called the tertiary wall has lately been the object of intensive investigations. Bucher (*17*) has studied this layer in softwood tracheids with a special staining technique. As an essential result of his work Bucher states that the innermost part of the cell wall is a highly individualized layer, clearly differentiated from the S_2-layer. Meier (*6*) investigated the S_3-layer in birch by studying the resistance of this layer against fungal attack. He found that it was very resistant and that it could be compared with the primary wall in that respect. Meier states that this resistance is probably a consequence not only of the protecting effect of incrusting meterial present in S_3 but also of the special physicochemical properties of the microfibrils in S_3. Thus microfibrils from S_3 though completely free from incrusting substances are more resistant against fungal attack than those of S_2. In the birchwood S_3 is free from lignin, which does not seem to be the case for softwoods.

The question of the chemical composition of the microfibrils in this layer must be left open at present. Meier indicates that xylan (in the form of incrusting material or microfibrils) might be an important component in the S_3-layer of birch.

The direction of the microfibrils of S_3 in birch and spruce seems to be nearly parallel to the fiber axis, which explains that no double refraction in a cross section is shown. Methods based on polarization studies alone are therefore not sufficient to study S_3 in birch and spruce (Meier). In other genera than *Picea* and *Betual* the fibrils in S_3 seem to be organized in flat helices. On the surface of S_3 toward the lumen the nonfibrillar material sometimes forms grains or warts, which is the case of birch. The same surface of the sprucewood tracheid, on the other hand, does not show any indications of such a submicroscopic structure.

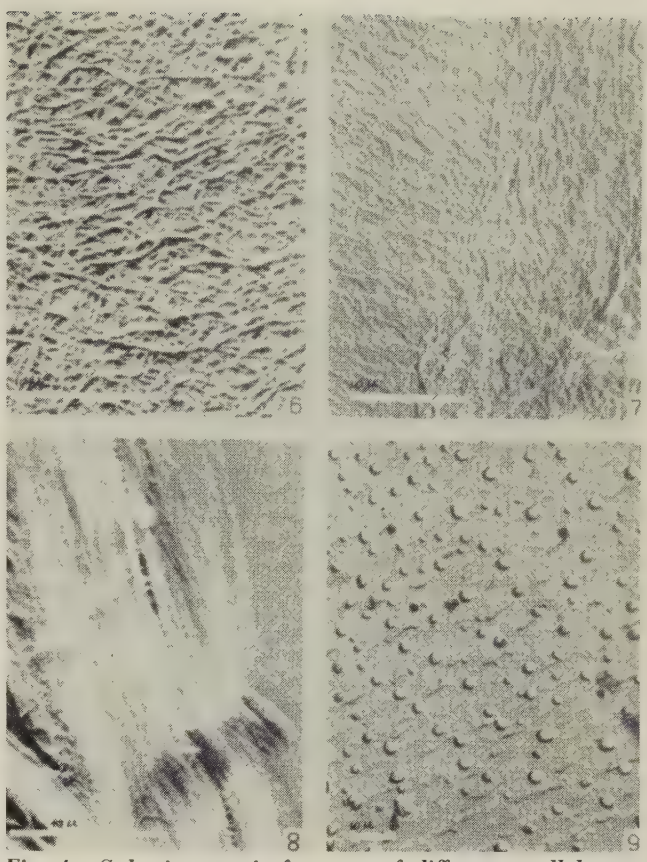

Fig. 4. Submicroscopic features of different wall layers (after Frey-Wyssling (11))

Upper left—Primary wall of the cambium-dispersed texture (photographed by H. H. Bosshard). Upper right—Transition layer-crossed texture. Lower left—Secondary wall-parallel texture with "slip plane." Lower right—Warts on tertiary wall.

In Fig. 4 electron micrographs show the different types of the fibrillar organization in the primary wall and the layers S_1 and S_2 together with the warts on the lumen surface of S_3.

Figure 5 schematically shows the structure of a tracheid or a fiber. Since the fibrillar direction in S_3 appears to vary within wide limits no principal direction of the fibrils is shown in the figure.

The terminology in connection with S_1, S_2, and S_3 was recently thoroughly discussed between Wardrop and Dadswell (*9*) and Meier (*18*). The former authors prefer to keep the older terminology of Kerr and Bailey.

The present somewhat unclear situation concerning the character of S_1 and S_3 calls for more fundamental work, from both a physical and chemical point of view, on the fibrillar and nonfibrillar matter in these layers.

Modifications in the Cell Wall

During early stages of the thickening of the primary wall thin areas are left uncovered. As thickening pro-

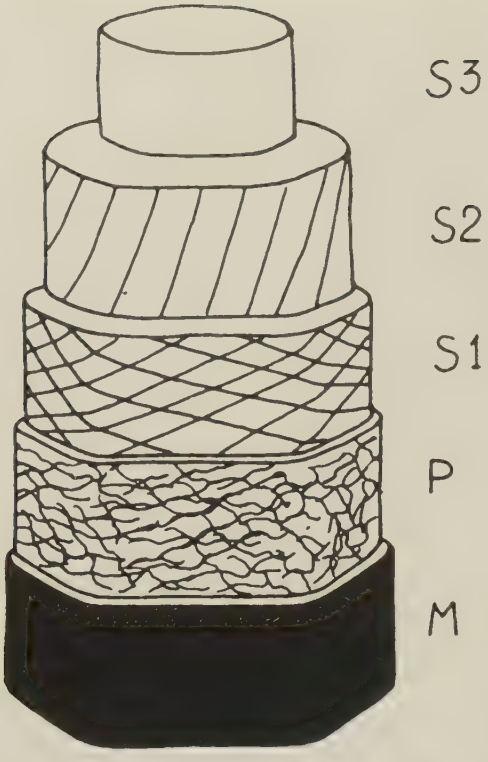

Fig. 5. The fibrillar organization of a fiber or a tracheid

ceeds cavities are left in the wall at these places. The wall around the cavity, the cavity and the primary wall membrane are called a pit. Pits occur as pit-pairs on both sides of the primary wall of contiguous cells. They are characteristic of all kinds of cells. They are abundant in cells for conduction as, for instance, in vessels and springwood tracheids. They show great variations in type and form. They are of a certain technical importance from the impregnation point of view. Especially the fine structure of the pit membrane is of interest. Few investigations have been made on the fine structure on pit membranes from hardwood. The most recent one (*19*) shows that the microfibrils in the membrane are arranged in the same way as in the primary wall. It has no torus like the pit membrane in softwood tracheids and there are no openings of microscopic size in the membrane. The microfibrils appear to be enveloped in incrusting substances.

THE DISTRIBUTION OF THE COMPONENTS IN THE CELL WALL

It would be of considerable interest to have further information on the ultramicroscale about the distribution and organization of the different chemical components across the cell wall. From electron microscopical investigations of the different layers of the cell wall associated with chemical analysis of the microfibrils and the less ordered material it seems possible to gain more extract information on this important problem. However, the technical difficulties which are met with here are almost overwhelming due chiefly to the small dimensions of the object and consequently to the small amounts of matter. For a reliable chemical analysis of a certain layer in the cell wall it is necessary to isolate a sufficient amount of that layer, which seems to offer considerable difficulties.

However, on a bigger scale, i.e., with a resolution of about 0.5μ some quantitative or semiquantitative microoptical determinations have been carried out on the distribution of "cellulose," "hemicellulose," and lignin in the cell wall of spruce and birch fibers (*20*). It must be observed that these results cannot always be directly compared with those obtained with the aid of the electron microscope owing to the different scale of resolution. For instance the thin primary wall in a cross section of the cell wall cannot be quantitatively investigated in the light microscope, as is probably also the case with S_3. It is therefore highly probable that future quantitative measurements of the distribution of the component on the submicroscopic scale will change, modify or at least give further and more detailed information on how the components are deposited in the cell wall. At present the situation can be described in the following way.

Measurement of the absorption of radiation in the ultraviolet range in cross sections of wood show that lignin is mainly deposited in the compound middle lamella. Hence, the average concentration of lignin in the other layers of the cell wall must certainly be considerably below the average concentration of lignin in wood, i.e., about 30% for spruce and about 20% for birch. The lower content of lignin for birch is also valid for other kinds of hardwood as, for instance, ash, aspen, beech, and elm. Further, measurements show that the great change in lignin concentration in the cell wall takes place within S_1.

Outside this layer the cell wall can be considered as composed mostly of lignin. Inside S_1, where the major part of the cell wall is localized, on the other hand, the carbohydrates are the dominating components. This result indicates that it is highly probable that there is a great change in fiber properties in the narrow range of the S_1 layer. Part of S_1, S_2, and S_3 are thus roughly the carbohydrate part of the fiber and the compound middle lamella and the rest of S_1 the lignin part.

It is supposed that lignin gives wood certain mechanical properties. If so, differences between the lignin distribution in normal wood and reaction wood could be expected.

A comparison between tension wood and normal wood from red beech shows that the cell walls of normal wood contain more lignin than those of tension wood (*14*). In the later case the lignin is deposited almost entirely in the compound middle lamella with the cell walls practically free of lignin. The normal division of the inner cell wall into three layers is often replaced by two, three, or four layers (Fig. 6) The innermost layer, the so-called gelatinous layer, which thus can be S_2, S_3, or S_4, is completely free from lignin.

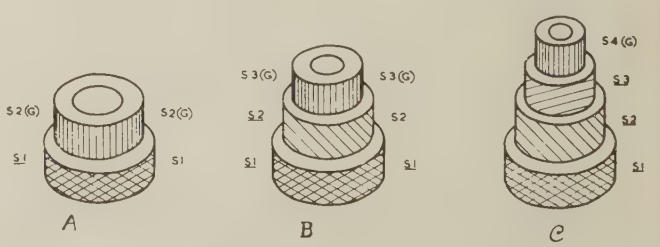

Fig. 6. Diagrammatic representation of the organization of the secondary wall in various types (A, B, C) of tension wood. G = gelatinous layer (after Dadswell, Wardrop, and Watson in (10))

It should be observed that tension wood occurs fairly frequently. Hence, it is also of technical importance to know the morphology of this special kind of wood (*10*).

From microspectrographic measurements on the distribution of carbohydrates across the cell wall it can be stated that:

1. Holocellulose is approximately evenly distributed across the cell wall in spruce and birch. In the latter case there is perhaps a tendency toward an increasing amount of carbohydrate material toward the outer regions of the cell wall.

2. "Cellulose" is most densely packed around the lumen in spruce and birch with the relative packing density in the outermost layers roughly about half of that around the lumen. This seems to be verified on the submicroscopic scale by the observations of Wardrop and Dadswell (*9*) that the density of lamellas in the fiber wall of *Eucalyptus goniocaly* increases toward the lumen.

3. "Hemicellulose" is about half or more of the carbohydrate material in the outer regions of the cell wall of spruce and birch. The "hemicellulose" content in S_2 for birch seems to be somewhat higher than that for spruce. Around the lumen the percentage of carbohydrate material consisting of "hemicellulose" is estimated at 10 to 20%.

Measurements of the absorption of soft x-rays in different layers of the cell wall (*21*) can be interpreted in such a way that the compound middle lamellas has a more porous structure than S_3 in the case of both spruce and birch. Further the results indicated that S_2 in birch has a somewhat more porous structure than S_2 in spruce.

Asunmaa and Steenberg (*22*) in their quantitative measurements on electron scattering could not find a similar difference in porosity for hardwood of spruce and pine. As a matter of fact, they found the opposite effect. However, their conclusions are based on the assumption that there is no significant difference between the concentration of inorganic matter in the middle lamella and the secondary wall, contrary to the result of Lange (*23*), who, using microincernation methods, could show that most of the inorganic material in the tracheids of Swedish spruce was deposited in the compound middle lamellas.

FIBER MORPHOLOGICAL FACTORS AND THE PROPERTIES OF PULP, PAPER, AND THEIR DERIVATIVES

The properties of the products made from wood are obviously more or less related to the morphology of the wood fibers. In an oversimplified manner this dependence can be described in terms of two factors, namely, the shape of the fiber and the structure of the fiber wall. The latter factor is often referred to in terms of, for instance, crystalline-amorphous, order-disorder, accessible-inaccessible, etc., in relation to the general term of reactivity. For a detailed review the reader is referred to the literature (*7, 8*).

Work carried out in this field has, naturally enough, mainly been associated with the morphological properties of the softwood tracheids. A rough picture of the influence of the shape of the hardwood fibers on, for instance, the properties of paper can be obtained if one remembers that the abundant hardwood fiber material consists of thicker walled, rigid fibers. With respect to their appearance in paper they can to some extent be compared with the thicker walled summerwood tracheids. Contrary to thinner walled springwood tracheids they keep their tube form during beating and do not collapse into ribbonlike entities.

Previously it was thought that the shorter hardwood fibers would result in lower tensile strength. However, new types of hardwood pulps show tensile strength of the same order as softwood pulps. Other mechanical properties of hardwood paper are generally not so good as for paper made from softwood pulps, owing to the shorter fiber lengths of the hardwood fibers. For the same reason, on the other hand, hardwood fibers generally form a more uniform sheet, the opacity of which is very good, owing to the great ratio of outer fiber surfaces to fiber weight.

Since the general structure of the hardwood fiber wall is the same as that of the tracheid wall it is to be expected that the relation between fiber wall structure and, for instance, pulp quality would be similar for hardwood and softwood fibers. It should be observed, however, that much remains to be done in this field mainly owing to the obvious fact that complete knowledge of the fiber structure on the ultramicroscale is still lacking.

There is a definite difference in lignin distribution across the fiber wall between hardwoods and softwoods, the cell wall of the former being less lignified.

Difference in behavior during cooking and bleaching of hardwoods and softwoods are partly due to this difference in lignification. For instance, a sulphate pulp from birch can be defibrated only at a Roe number of 3 to 4, while a similar pulp from spruce is defibrated at a Roe number of about 8. On the other hand, the birch pulp is easier to bleach, since most of the lignin is easily accessible.

When beating wood fibers, the removal of the primary wall and the swelling, in as much as it makes the fiber more flexible, seem to be the main effects in improving paper strength according to Giertz (*10*). The primary wall having a more or less isotropic plywood structure will not swell, contrary to the other cell layers, where the fibrils are more parallel to each other.

Emerton emphasizes the great influence of the structure of S_1 on the beating process (*10*).

The S_1 and S_3 layers are said to be less "reactive" in connection with the viscose process. Fragments from these cell wall structures have been found (*7*) in technical viscose solution. These results are similar to those of Meier (*6*) who found that the microfibrils of S_1 and S_3 were very resistant against fungal attack.

Finally, it should be mentioned that pulp from tension wood is said to have very poor papermaking properties owing to the lower pentosan content in the tension wood fiber wall. Cell wall deformations formed during the development of tension wood are a source of weakness in pulps prepared by acid pulping methods. On the other hand tension wood appears to produce mechanical pulps superior to those from normal wood (Dadswell, Wardrop, and Watson in *10*).

THE MICROFIBRIL UNIT

The study of the smallest morphological unit so

Fig. 7. Electron micrograph of micro fibrils from animal cellulose (Ranby)

far, the microfibril (Fig. 7), seems to be of the greatest interest in the field of future fiber structure (Ranby, in *10*). It is highly probable that further research in this field will also be of the greatest technical importance.

Electron microscopy of thin sections of plant cell walls have given good evidence that cellulose and probably also certain fractions of hemicelluloses are deposited in the fiber wall in the form of more or less ordered microfibrils, appearing as flat ribbons of about 100 A width and of indefinite length. The width/thickness ratio seems to be 3 to 4. A schematic cross section of a bundle of cellulose fibrils in a secondary plant cell wall is shown in Fig. 8. There is also electron microscope evidence that hemicellulose and lignin is dispersed between the fibrils through the whole fiber wall. There seem to be slight differences in fibril widths between cellulose fibrils of different origin. Animal cellulose appears to contain somewhat wider fibrils (100 to 150 A) than do cotton, ramie, and hemp cellulose (90 to 100 A) and cellulose from wood (80 to 100 A). Jayme and Koburg (*24*) have reported significant differences in widths between fibrils from different beech fibers and those from softwood tracheids. However, the order of width is far greater than those shown above. Further work in this field appears to be necessary.

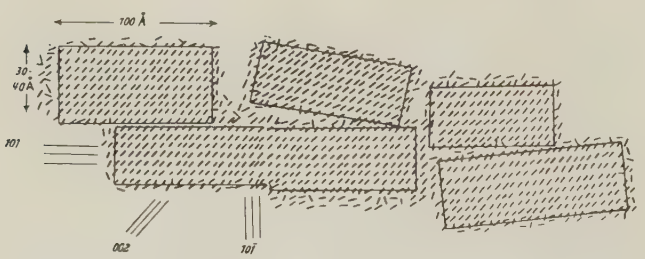

Fig. 8. Schematic cross section of a bundle of cellulose fibrils in a secondary plant cell wall—101 planes of the lattice are parallel with the plane of the cell wall (after Ranby (10))

The lattice order of the cellulose microfibrils depends upon the biological origin of the fibrils. From x-ray measurements, swelling, and hydrolysis experiments it can be stated that fibrils of wood, cotton, and animal origin show improved lattice order as mentioned (Ranby, in *10*, *25*). This difference in the inner structure of the cotton and wood cellulose fibrils is of great technical importance. Many differences in chemical behavior ("reactivity") between cotton and wood celluloses appear to be more related to this latter difference in lattice order of the microfibril than to differences in the gross structure of the fiber. Sufficient information about eventual differences in lattice order of fibrils from different wood origin (softwood contra hardwood, reaction wood contra normal wood) is lacking.

It is probably not saying too much if one assumes that the ultimate reason for the difference in lattice order is the difference in the conditions of formation and growth of the microfibril. An interesting example supporting this assumption is the difference in lattice order between the microfibrils from the cotton stalk and those from the cotton seed hair. The former type of microfibril is not a cotton cellulose fibril. It must be considered as a wood cellulose fibril. Thus the plant origin as such has no influence upon the character of the microfibril.

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Chemical Aspects of Hardwood Lignins

J. M. PEPPER

The present knowledge regarding the chemical structure of the protolignin of hardwoods is reviewed. The methods and problems associated with its isolation are discussed. For hardwoods, any representative lignin fraction, mainly comprised as it is of both guaiacyl- and syringyl-containing nuclei, presents added problems of homogeneity and chemical alteration during isolation. The research that has been directed towards the elucidation of lignin structure has been conducted using woodmeal directly or using an already isolated lignin which undoubtedly has undergone some chemical change. Considering these two approaches, the more important and older fields of study, such as: solvolyses, hydrogenation and hydrogenolyses, oxidation and alkali fusion; as well as the more recent approaches of biogenetic and biosynthetic studies using C^{14}-labelled precursors, and ultraviolet and infrared absorption spectra, are discussed in the light of their individual contributions to the solution of the lignin problem. An attempt is made to correlate the more important of these findings and to present a composite picture of the present status of the chemical structure of hardwood lignin. The problem remains far from solved and some of the major questions yet to be answered are raised, along with suggestions to paths that future research may profitably take to assist in their solution.

THE object of this review is to present, in a general way, the present status of the knowledge of hardwood lignins. No attempt will be made to discuss in detail all recorded researches in this field. Rather it is hoped that a discussion of the significance of the recorded data, with particular emphasis being placed on those aspects of the problem wherein further research is indicated, may be of some value to those interested in the field of hardwood lignin chemistry.

After having surveyed the work that has been done by the many investigators who have been interested in lignin, any attempt to define this common but challenging natural resource recalls to mind the Hindoo fable of "The Blind Men and the Elephant," as written by J. G. Saxe.

"It was six men of Indostan
To learning much inclined,
Who went to see the Elephant
(Though all of them were blind),
That each by observation
Might satisfy his mind.

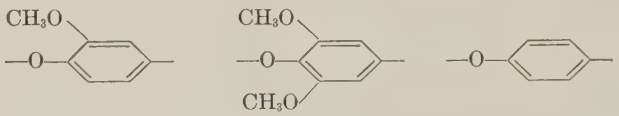
. . . . . .

And so these men of Indostan
Disputed loud and long,
Each in his own opinion
Exceeding stiff and strong,
Though each was partly in the right,
And all were in the wrong."

It is not being suggested that the plant anatomist, the plant physiologist, the plant biochemist, the chemist or the pulp and paper engineer are blind, nor that they are all in the wrong; but to each lignin presents a different challenge. It is becoming increasingly evident that it will be only by the combined efforts of all that the mysteries of lignin will be solved.

From the viewpoint of the organic chemist all mature woody plants contain a chemical substance, lignin, which is distinctly different from the other major components of cellulose and hemicelluloses. Being a biological product it is natural to expect some variations in its occurrence and chemical composition. Herein lies one of the difficulties still confronting the lignin chemist. It is rather readily proved, by analytical degradation studies to be discussed later, that there is a marked difference between the lignin of angiosperms (hardwoods) and gymnosperms (softwoods), but there are also smaller though significant variations in chemical composition of the lignin within these two major classes. As a result of extended experiments, Pearl (*1*) reports that the nature of the lignin of aspen does not agree with traditional concepts of lignin—even deciduous or hardwood lignin. Some of these observed variations are undoubtedly the result of the varied lignin isolation techniques—but there is no means at present of proving that others are not inherent in the protolignin of the plant.

The lignin of angiosperms, containing as it does varied amounts of para–substituted-*o*-methoxyphenoxy- (*A*), para–substituted-*o*,*o*′-dimethoxyphenoxy- (*B*) and para–substituted phenoxy- (*C*) nuclei containing portions, presents added problems over those associated with softwoods lignin which never have been shown to contain any but very small amounts of the latter two nuclei (*2*, *3*).



A B C

There is therefore a much greater number of possible ways of integrating the units of hardwood into a lignin substance than there is for softwoods. This proposed chemical nonhomogeneity of the lignin substance of angiosperms would suggest that a study of this material would be much more complicated than that of softwoods. By means of electrophoretic and paper chromatographic studies Nord and Schubert (*4*) have reported that both native and enzymically liberated samples of oak and birch lignin behave as homogeneous preparations whereas similar preparations from maple do not. Indeed, one of the major unsolved problems respecting hardwood lignin concerns the nature of the periodicity of these aromatic units. There is no positive indication whether lignin is either a discreet mixture of polymeric units of each basic type as suggested by (I), or whether it is a highly complex polymer of all

J. M. PEPPER, University of Saskatchewan, Saskatoon Sask.

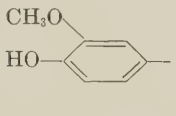
basic types joined with or without any definite repetitive pattern (II).

$$\begin{bmatrix} \text{-A-A-A-A-} \\ \text{-B-B-B-B-} \\ \text{-C-C-C-C-} \end{bmatrix} \qquad -(A)_x-(B)_y-(C)_z-\text{etc.}$$

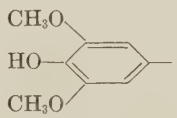
Type I Type II

Neither can the possibility of crosslinking among these polymeric chains be overlooked. No significant success has been achieved in effecting the separation of lignin fragments of any one of these distinct types. Pearl and Beyer (*5*) have reported that isopropanol fractionation appeared to fractionate the lignosulfphonates from aspen spent liquor on the basis of syringaldehyde- and vanillin-yielding moieties.

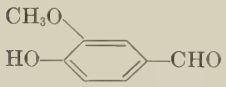
On the other hand the study of at least a part of angiosperm lignin may be simplified by the fact that the syringyl-type compounds which arise from the type B nuclei have both positions ortho to the phenolic hydroxyl grouping blocked by methoxyl groups.



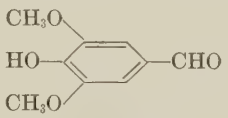
Guaiacyl radical



Syringyl radical



Vanillin (III)



Syringaldehyde (IV)

It is well known that the free 5-position of guaiacyl compounds is reactive and that either during lignin formation itself or as a secondary reaction during isolation, inter or intramolecular condensation may occur at this center. This is not possible with the syringyl type, and thus for lignin containing such nuclei the possibilities of union are reduced. The usual greater percentage yields of syringaldehyde (IV) over vanillin (III), as a result of the alkaline nitrobenzene oxidation of angiosperm substances, may be a result of this fact. It is not yet known if the relative proportions of such isolated aldehydes may represent similar proportions of these nuclei in the plant. This will be discussed further.

It has been inevitable that much greater attention has been given to the chemical composition of softwoods lignin and hence much more information is available on this type than there is on hardwoods lignin. Realizing that this latter lignin in part appears to be similar (as a precursor of guaiacyl nuclei derivatives) and undoubtedly biogenetically closely related, to the former type, the hardwoods lignin chemist has the opportunity to utilize this information. In as much as there is a marked chemical difference between these lignins, there is no logical reason for believing that those portions of the macromolecule which appear to be common to both, need necessarily be identical. For the purposes of this review present ideas regarding the chemical composition of hardwoods lignin will be presented therefore on the basis of experiments involving hardwoods only.

A discussion of the structure of lignin may be divided conveniently into four parts;

A. Information obtained as a result of the isolation and subsequent chemical of physicol-chemical investigation of a lignin substance.

B. Information obtained as a result of the degradation of protolignin or isolated lignins and the subsequent isolation of smaller molecular weight, identifiable products.

C. Information deduced as a result of biogenetic studies using model substances as lignin precursors and radioactive tracer techniques.

D. Information deduced as a result of miscellaneous observations particularly using lignin model compounds.

A. THE ISOLATION OF LIGNINS

Two important considerations that arise in studying the isolation of any natural product are (1) the yield and (2) the extent of chemical alteration during isolation. For the hardwoods lignin chemist the first requirement has added significance. If, as it has been suggested, the lignin may not be homogeneous, it is difficult to know if any isolated fraction is representative of the lignin as a whole. The situation readily arises that the conclusions drawn from one experiment, though valid, may not agree with those drawn equally validly from a separate experiment. It is interesting to point out that in a study of hardwoods lignin a novel method of comparison presents itself which is not available for softwoods lignin. The ratio of the different nuclei (A, B, and C) as determined by the isolation of nuclei-containing fragments may be used to compare isolated lignins. In Table I are presented the data on the isolated lignins of representative hardwoods. With the exception of those procedures involving alcohols in which some etherification does occur, only those methods of isolation are indicated which may be considered to yield a lignin fraction either as it pre-existed in the plant or as a result of inter or intramolecular reaction with itself or other plant components.

Table I. Isolated Lignins of Representative Hardwoods

Nature of Lignin	*Wood*	*Yield,*[a] *%*	*Analyses, %*			*Ref.*
			OCH_3	*C*	*H*	
Native	Aspen	0.7[b]	19.8	63.5	6.0	*6*
	Oak	...	14.8	58.6	5.3	*7*
	Birch	...	14.9	61.4	5.5	*7*
	Maple	...	17.4	61.0	5.6	*7*
Enzymically liberated	Oak	...	14.6	58.4	5.2	*7*
	Birch	...	14.8	61.6	5.6	*7*
	Maple	...	17.8	61.3	5.5	*7*
Klason	Aspen	100	20.4	...	...	*8*
	Beech	100	17.4	62.1	5.0	*8*
Willstätter	Aspen	...	17.6	63.2	5.4	*8*
	Beech	...	14.5	64.5	5.9	*8*
Cuproxam	Beech	13	22.2	60.2	5.8	*8*
Alkali	Aspen	...	...	63.3	5.8	*8*
	Birch	...	...	62.2	5.5	*8*
	Maple	[c]	14.1	62.4	5.5	*8*
Periodate	Maple	22.3	20.4	58.4	5.4	*9*
	Birch	21.8	21.4	57.6	5.3	*9*
	Beech	24.4	16.7	54.6	6.1	*9*
Björkman	Aspen	...	21.5	...	...	*10*
	Birch	...	21.5	...	...	*10*
Hydrol						
Ethanol	Maple	70–80[d]	24–25	...	...	*11*
Dioxane (alkali)	Maple	104	...	...	...	*12*
Dioxane-water	Aspen	95	21.6	...	...	*13*
Solvolytic						
Methanolysis	Beech	22.5	25.5[e]	...	...	*14*
	Aspen	20.4	25.6[e]	...	...	*14*
Ethanolysis	Maple	80	...	...	...	*15*
Dioxanolysis	Beech	19.0	21.	62.5	5.5	*16*
	Aspen	(see Tables II and III)				
Liquid ammonia	Maple	6	21.6	58.2	6.4	*18*
Hydrotropic	Aspen	65	17.4	61.5	5.4	*19*

[a] Yields reported as percentage of Klason lignin.
[b] Molecular weight, 945 (*6*).
[c] Molecular weight, 4500–10,000 (*20*).
[d] Molecular weight, 905–1915 (*20*).
[e] High methoxyl content due to etherification by methanol.

An examination of Table I cannot but emphasize the problem of variation in analytical data and especially the need for caution in the interpretation of such data in the light of the yields obtained. There is only general agreement on the methoxyl contents of such lignins, even for one species, e.g., aspen, the reported values range from 17.4 to 21.6%. It should also be remembered that all wood meals are solvent-extracted to remove extraneous components and often this procedure varies from worker to worker. What effect such prior treatment may have on the subsequent isolation of lignin has not been determined. Recently, Pearl (*1*) has again questioned the status of Klason lignin as it is used as a measure of the lignin in wood. In a study of the methoxyl balance made on a Klason lignin determination of aspen wood meal, although 92.5% of the original methoxyl was recovered, the solid Klason lignin contained only 65% of the original content.

The importance of the conditions of the isolation procedure must not be underestimated; the yields and composition of isolated lignins depending on such factors as temperature, time, pH, and the nature of the suspending medium. For this reason it is very difficult to even compare the results in Table I. As a particular example of this variability may be cited the results obtained in the author's laboratory. A study has been made of the isolation of aspen lignin by means of an acid-catalyzed solvolysis in a dioxane-water (9:1) solvent system. The results are shown in Tables II and III.

Table II. Effect of Time on Aspen Lignin Isolation (*17*)[a]

Reflux time, hr.	*Isolated Lignin,[b] %*	*Analyses*				
		OCH_3, %	*Oxidation products, % Syringaldehyde*	*Vanillin*	*Ratio S/V*	*C-CH_3/ OCH_3*
0.5	42.2	21.0	23.2	8.13	2.86	0.13
1.0	54.3	21.0	23.9	8.72	2.74	0.11
2.5	68.8	20.8	17.8	6.38	2.79	0.11
4.0	71.0	20.3	13.8	4.74	2.91	0.15
8.0	76.3	19.5	10.5	4.65	2.26	0.22
48.0	107.0	16.3	5.6	3.71	1.51	0.29
Wood meal	100	...	33.8	16.6	2.04	...

[a] Initial hydrochloric acid concentration 0.2 *N*.
[b] Reported as per cent of original Klason lignin.

These data support the belief that lignin isolation is essentially a two-stage process: a hydrolysis stage followed by dissolution in a good lignin solvent system. It would appear also that there is an early rapid rate of isolation after which the rate slowly declines as does the methoxyl content of the lignin fraction so isolated. This suggests that two basic types of lignin linkage are involved, and only after the major part of the more easily hydrolyzed fraction is removed does the second type become prominent. Earlier, in a very interesting study of the dissolution of aspen wood using neutral buffered butanol-water solutions, Aaltio and Roschier (*22*) presented evidence for a similar break in the isolation curve. Their assumption was that in aspenwood, the lignin forms with pentosans, two different complex compounds with the stoichiometrical ratios L:P 0.85 and L:P 0.64, and which have methoxyl contents of 20 to 21 % and about 15%, respectively.

It is also clear from the analytical results of Tables II and III, that the chemical nature of the isolated lignins changes markedly during isolation and that methoxyl content is of little value as a basis of comparison. The

Table III. Effect of Acid Concentration on Aspen Lignin Isolation (*21*)[a]

HCl conc. N.	*Isolated lignin,[b] %*	*Methoxyl content, %*
0.00	1.61	...
0.055	17.1	...
0.11	22.2	20.6
0.22	44.0	20.7
0.40	62.9	20.7
0.80	74.8	20.8
1.69	89.6	18.9

[a] Time of reflux, 0.5 hr.
[b] Reported as per cent of original Klason lignin.

steady decrease in the yields of the phenolic aldehydes, vanillin (III) and syringaldehyde (IV), may be explained in many ways. It may be due to (1) the increased isolation of a lignin fraction whose structure never was capable of being oxidized to these derivatives; (2) chemical change before or during the solvolytic cleavage, or (3) chemical change occurring as a result of acid-catalyzed reactions once the lignin has been solubilized.

The Lignin-Carbohydrate Complex

Undoubtedly one of the *major* considerations of lignin isolation is that of the existence or nonexistence of a lignin-carbohydrate complex. Recently, more and more evidence has been presented in support of a definite lignin-hemicellulose union in the wood. Björkman (*10*) in a study of the lignin-carbohydrate complexes of birchwood, isolated as his milled wood lignin, showed that after complete hydrolysis of the isolated complex, the kind and amount of the individual monosaccharides obtained agreed well with those resulting by a similar hydrolysis of the hemicelluloses from the same wood. Pew (*23*) in a study of aspen lignin isolation by cellulytic enzymes found evidence for a lignin-carbohydrate linkage in wood. When powdered wood dissolves in aqueous sodium thiocyanate, the carbohydrate carries the normally insoluble lignin into solution and when an impure enzyme lignin is dissolved in a solvent, such as benzyl alcohol, the carbohydrate dissolves because it is attached to the lignin. Pew suggests, "it appears that complete and quantitative separation of lignin from carbohydrate by means of enzymes may not be achieved . . . the type of association between the lignin and wood remains speculative." The nature of these isolated lignins described in Tables II and III can best be considered as containing increasing percentages of carbohydrate material. This would account for the decreasing methoxyl content and the decreasing yields of aldehydes on oxidation. Similar reasoning has been advanced (*13*) to explain those yields (based on chloroform solubility), which are obtained by the hydrogenolysis of aspenwood and which are greater than 100%.

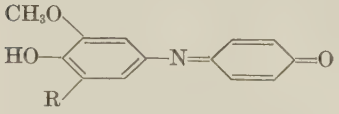
Analytical Studies of Isolated Lignins

It has been customary to obtain as much analytical data as possible on any isolated lignin. This permits of the assigning of a tentative lignin molecular formula and the opportunity to compare these data with related data obtained from a study of lignin model substances. In this category fall molecular weight studies, fundamental elemental and functional group analyses including hydroxyl (both aliphatic and phenolic), carbonyl, alkoxyl, end-methyl, and unsaturated groups as well

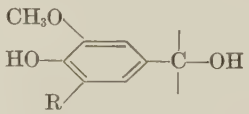
as the ultraviolet and infrared absorption spectra studies. For hardwood lignins such analytical data are not so complete as for softwood lignins. This fact considered in relation to the two factors already discussed, variable yields and possible nonhomogeneity, make it imperative that these data be interpreted with considerable caution.

Being of similar nature to other naturally occurring polymeric materials, any average molecular weight values for isolated lignins are dependent on the methods used for such determinations. The problems associated with such determinations have been discussed by Schuerch *et al.* (*20*) who point out that in the very range that covers the average molecular weight of isolated lignins (300 to 5000) the precision of the more common methods is not great. These authors recommend the use of the cryoscopic method in ethylene carbonate over a similar method using dioxane or an isopiestic method in tetrahydrofuran. Some molecular weights have been reported in Table I.

Considerable attention has been given to the phenolic hydroxyl content of softwood lignins but much less to that of hardwood lignins. Sarkanen and Schuerch (*24*) have discussed the various methods available. By a conductometric procedure they determined the equivalent weights of maple ethanol lignin, aspen native lignin and maple hydrol lignin to be 422, 455, and 744, respectively. The presence of parahydroxyphenyl carbinol groups in wood lignin has been proved by an interesting reaction reported by Gierer (*25*). The reaction of quinone monochloroimide with aspen wood meal with the subsequent isolation of both 2-methoxyphenolindophenol (V) and 2,6-dimethoxyphenolindophenol (VI) was taken as proof of the presence of units of the types (VII) and (VIII), respectively.

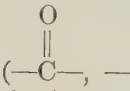


V: R = H
VI: R = OCH_3



VII: R = H
VIII: R = OCH_3

Both ultraviolet and infrared absorption as well as electrophoretic studies on isolated lignins have been, for the most part, of greatest value as a means of comparison, of either lignin to lignin or lignin to model substances. They have contributed less to the knowledge of fundamental structure. The absorptivity at wavelengths thought to be characteristic of lignin (around 280mμ is characteristic of the aromatic nuclei) is being extensively used as a quantitative estimation for lignin. Considerable attention has been given to the probable presence of unsaturated systems



(—C—, —CH=CH—) conjugated with the aromatic ring but no definite conclusions may be drawn. It is significant that Nord, *et al.* (*4*), report that the native lignins of oak, maple, and beech have clear cut maxima at 280 mμ, whereas aspen native lignin shows no such maximum. Recently more attention has been given to the infrared studies in an attempt to identify some characteristic chemical features. The difficulties in preparing suitable lignin samples in the form of liquid or solid solutions, films, or mulls have added to the problem. On the basis of known chemical differences between hard- and softwood lignins, one might expect essentially different spectra. However, it has been reported (*4*) that the infrared spectra of birch, oak, and maple native lignins are similar and indeed similar to that of a typical softwood lignin from white Scots pine. Absorption bands at 1600 cm.$^{-1}$ and 1510 cm.$^{-1}$ have been ascribed to the aromatic nature of lignin. Rather prominent bands at 1725, 1430, and 1325 cm.$^{-1}$ have been noted and attributed to ester or acid carbonyl and aliphatic groupings. On the basis of some rate studies on the hydrolysis of native lignin and several model compounds, and on the isolation of parahydroxybenzoic acid, Smith has proposed (*26*) that aspen lignin contains parahydroxybenzoate groups esterified with aliphatic hydroxyl groups of lignin. In our laboratory the infrared spectra of the aspen lignins isolated by the acidolysis procedure (Table II) were found to be identical and resembled very closely the spectrum of a sample of aspen native lignin. In as much as the yield of aldehydes obtained by the oxidation of these lignins were markedly different, it is suggested that the use of infrared spectroscopy for such an exceedingly complex substance may not be of particular value at least as regards fine structure. On the other hand the spectra of the lignins isolated at various acid concentrations (Table III) showed marked differences: the absorption at 1600 cm.$^{-1}$ decreasing with increasing pH and the absorption originally at 1510 cm.$^{-1}$ shifting to higher wave numbers. Wide absorption bands indicated the presence of hydroxyl groups in all preparations.

B. THE DEGRADATION OF LIGNIN

With respect to the fundamental chemical composition of liginins, a rather more productive aspect has been the studies dealing with lignin degradation. By various means, lignin, either in an isolated state or still as part of the complex cell components in wood meal itself, has been degraded chemically to produce units which are capable of being isolated, purified, and characterized. Studies of the yields and the chemical composition of these lignin derivatives as well as the conditions under which they were isolated, have contributed most to what is presently known about lignin structure. In no such study has it been found possible to identify all the lignin (on a carbon basis) as smaller units. It should be emphasized that because of the relatively small recoveries in most cases, there is no way of knowing if the derivatives obtained by one procedure originate from the same part of the lignin polymer as do those obtained by another technique. Studies using radioactive lignin precursors may provide some information in this connection and should prove of value in determining the relative ease of isolation of each particular unit. Neither can it be assumed that the nature of these products isolated bears any relationship to the unidentified lignin residue. Efforts should be directed continuously toward the characterization of greater percentages of lignin degradation products. Significant contributions have been made through studies of the processes of alkaline hydrolyses, oxidation, acid-catalyzed solvolysis, and hydrogenolysis.

Alkaline Hydrolysis

A detailed study of the alkaline hydrolysis of representative hardwoods has been made by Pearl and co-

workers (*27*, *28*). The ether extract of the acidified filtrate was chromatographed and a quantitative determination made of vanillin, syringaldehyde, *p*-hydroxybenzaldehyde, and vanillic, syringic, *p*-hydroxybenzoic, ferulic, and *p*-coumaric acids. Significant amounts of *p*-hydroxybenzaldehyde and/or the corresponding acid appear to distinguish aspen and willow from other species. The isolation of *p*-hydroxybenzoic acid from aspen by Smith (*26*) has already been mentioned.

Oxidation

In the early studies of lignin oxidations the characterization of relatively small percentages of aromatic products was not of major structural significance. This was rapidly changed with the development by Freudenberg of the alkaline nitrobenzene oxidation technique whereby isolated lignins or wood meal could be oxidized to give high yields of phenolic aldehydes. As a result of the refining of the method (*27*, *29*, *30*, *31*) for the isolation and quantitative determination of these aldehydes and related compounds, this technique now serves as both an analytical method and as a means of comparison of lignins, lignified materials, and lignin model substances.

The maximum yields of vanillin and syringaldehyde obtained by the alkaline nitrobenzene oxidation of solvent extracted aspen wood meal are 15.6 and 35.9%, respectively, based on the Klason lignin (*32*). On the presently assumed basis that lignin may be represented as a polymer of phenylpropane units and that the major part of the wood methoxyl is associated with the lignin, then this total yield of aldehydes represents only about a 60% recovery of lignin both on a carbon basis and on a methoxyl basis. No detailed information is available as to the remainder of the lignin although smaller amounts of closely related products of oxidation, acetovanillone, acetosyringone, *p*-hydroxybenzaldehyde and vanillic, syringic and *p*-hydroxybenzoic acids have been characterized.

A considerable number of unsolved problems are associated with these oxidation studies. The yields of the phenolic aldehydes so obtained by the oxidation of an isolated lignin are always considerably less than those obtainable from wood itself. This emphasizes the significant change that must occur in the structural elements of lignin during all isolation procedures. Aspen native lignin, on oxidation, gave rise to vanillin and syringaldehyde in only 10.2 and 20.0% yields, respectively (*13*), while aspen Klason lignin gave only trace amounts of these compounds. The dioxanolysis lignin, isolated after only a one-half hour treatment at 95 to 100°C. (*17*) (see Table II) yielded only 8.13 and 23.2% of these aldehydes. Nor did the residual pulp from this experiment yield any further significant amounts. An aspen hydrol lignin, isolated by a mild hydrogenolysis procedure (Raney nickel catalyst, initial hydrogen pressure 550 p.s.i.g., 5 hr. at 175 to 180°C.), and having a high methoxyl content of 21.6%, gave rise to these aldehydes in yields of only 3.37 and 14.4%.

A detailed study of the effect of temperature on the yields of syringaldehyde and vanillin obtained by the oxidation of aspen wood meal has been conducted (*32*). The maximum yield of each aldehyde was obtained at the same temperature. These maxima could be obtained by conducting the reaction at 170 ± 5°C. for 2.5 hr. or at 130 ± 5°C. for 13 hr. These results would indicate that similar types of linkages are cleaved to release each aldehyde and therefore if the rates of these reactions are similar then the ratio of aldehydes so obtained should be indicative of the ratio of these units in the lignin molecule. However the observed decreasing syringaldehyde to vanillin ratio with increasing temperature is contradictory.

The liberation of phenolic aldehydes by this oxidative process means that two processes, at least, are occurring. These are the processes whereby the free phenolic group is released and whereby the carbonyl group is established. Since it is highly probable that these two reactions occur independently, it should be possible to arrive at the same end result by means of two separate stage reactions. Treating aspen wood meal first with alkali, prior to the nitrobenzene oxidation, led to a marked, about 30%, decrease in the yield of aldehydes (*32*). Kratzl and Silbernagel (*33*) treated beechwood with water up to 200°C. for only 30 minutes prior to oxidation and showed that the aldehyde yield was decreased by 60% from that obtainable from untreated wood. In place of the aldehydes an insoluble polymeric phenolic substance together with an acid fraction were formed. It would appear that the over-all reactions resulting in the release of the stabilized phenolic aldehydes are competitive with others giving rise to further condensed substances which are no longer capable of being so oxidized. It may be concluded therefore that under solvolytic conditions the rate of this condensation reaction is high and explains the inability to obtain, from an isolated lignin, no more than 60% of the aldehydes obtainable from wood itself. Indeed it may be reasoned that these yields obtainable from wood meal are but net values and that the inability to recover, as mentioned earlier, no more than about 60% of the lignin in this fashion, need not be considered as evidence of lack of homogeneity.

Acid-Catalyzed Solvolysis

The original work by Hibbert and co-workers on the ethanolysis of wood and isolated lignins introduced a degradative procedure whereby significant yields of highly oxygenated propylphenolic compounds were isolated. As a result of a 48-hr. ethanolysis treatment of maplewood meal the lignin derivatives shown in Table IV were isolated in a combined total yield of around 10% of the Klason lignin.

Table IV. Ethanolysis of Maplewood (*34*)

Lignin derivative	*Yield, % of Klason lignin*	
R(R′)—CO—CH(OEt)—CH_3	3.1	(3.2)
R(R′)—CO—CO—CH_3	0.6	(1.9)
R(R′)—CH_2—CO—CH_3	0.05	(0.2)
R(R′)—CHO	0.10	(0.6)
Total (R-type)	3.85	...
Total (R′-type)	...	5.9

R = 4-hydroxy-3-methoxyphenyl.
R′ = 4-hydroxy-3,5-dimethoxyphenyl.

The total yield of identifiable products thus obtainable is small but a more significant contribution to lignin structure resided in the production of the propyl substituted derivatives highly oxygenated in the

Table V. Hydrogenolysis of Hardwood Lignin

Lignin source	*Catalyst*	*Temp., °C.*	*Compound isolated*	*Yield,[a] %*	*Ref.*
Aspen methanol lignin	CuCrO	255	4-*n*-propylcyclohexanol	11.5	*39*
			4-*n*-propylcyclohexanediol-1,2	3.8	
			3-(4-hydroxycyclohexyl)-1-propanol	25.0	
Maple ethanol lignin	CuCrO	250	4-*n*-propylcyclohexanol	8.1	*40*
			4-*n*-propylcyclohexanediol-1,2	1.9	
			3-(4-hydroxycyclohexyl)-1-propanol	3.3	
Maplewood	CuCrO	280	4-*n*-propylcyclohexanol, 3-cyclohexyl-1-propanol, 3-(4-hydroxycyclohexyl)-1-propanol	39.8	*41*
			4-*n*-propylcyclohexanediol-1,2	1.9	
Maplewood	RaNi	160–175	3-(4-hydroxy-3,5-dimethoxyphenyl)-1-propanol	8.8	*42*
			3-(4-hydroxy-3-methoxyphenyl)-1-propanol	0.84	*43*
			1-(4-hydroxy-3,5-dimethoxyphenyl)-propane	0.83	
Maplewood	RaNi (3% NaOH)	160–170	4-hydroxy-3-methoxyphenylethane	2.2	*44*
			4-hydroxy-3,5-dimethoxyphenylethane	15.4	
			4-hydroxy-3,5-dimethoxyphenylethanol	6.2	
Aspen wood	RaNi (3% NaOH)	160–170	4-hydroxy-3-methoxyphenylethane	2.0	*46*
			4-hydroxy-3,5-dimethoxyphenylethane	2.6	
			4-hydroxy-3,5-dimethoxyphenylethanol	0.37	

positions alpha and beta to the aromatic ring. In a study of the alcoholysis reaction Sarkanen and Schuerch (*35*) have shown that the per cent total oxygen is decreased and they explain this by assuming intramolecular condensation with loss of the elements of water. The larger yields of derivatives containing the syringyl nuclei agree with the results of other degradation studies. The absence of any C_6–C–C lignin derivatives under acidic contitions of isolation is structurally important since to date such units have been obtained only under alkaline conditions of either oxidation or hydrogenolysis (see later). More recently the isolation of these type substances has become important in C^{14}-labeled biogenetic studies since they represent purified compounds whose specific activities may be measured. The etherification that results during ethanolysis may in one sense be fortunate in that these derivatives are thereby stabilized and less likely to react further. On the other hand increasing attempts should be made to isolate solvolytic lignin products under conditions of minimum secondary reactions.

Another important observation that may be made regarding the products isolated by an acid-catalyzed solvolysis concerns the establishment of carbon-methyl groupings not originally present in the protolignin (*36*). Hibbert (*37*) suggested that these groups may arise as a result of a series of intramolecular side-chain rearrangements within either the lignin substance before cleavage or within the released propylphenyl derivative. This reaction has been discussed by Gardner (*38*) whose work on the ethanolysis of oxyconiferyl alcohol (3-hydroxy-1-(4-hydroxy-3-methoxyphenyl)-2-propanone) clearly proved the ready ease of such rearrangements. The determination of the rate and extent of formation of such carbon-methyl groups provides another method of studying lignin structure and represents a means of estimating the extent of alteration of any isolated lignin. This aspect is readily seen in the results outlined in Table II where, for the acid-catalyzed solvolyses of aspen lignin, a steady increase of carbon-methyl content occurs with increasing time of reaction. It is unknown if such rearrangements occur within the protolignin itself or only as a secondary reaction following partial hydrolysis of a lignin-carbohydrate complex whereby reactive functional groups may be liberated.

Hydrogenolysis

The third major procedure that has resulted in the isolation and characterization of significant amounts of lignin degradation products is that of hydrogenation and hydrogenolysis. Several early experiments conducted at high temperatures and pressures and over assorted catalysts led to the isolation of several aromatic and cyclohexyl compounds having one- or two-carbon side chains (*8*). The results of the more recent, successful hydrogenolysis of isolated hardwood lignin or hardwood meal itself are given in Table V.

The characterization of these compounds, especially the aromatic derivatives, has given much support to the propylphenyl nature of the lignin substance. Undoubtedly the most significant aspect is the ability to isolate, under strongly reducing conditions, those derivatives with oxygen positioned beta or gamma to the carbocyclic nucleus. It can only be inferred that such oxygen linkages preexisted in the lignin. Although by this procedure the yields of aromatic C_6—C—C—C compounds obtained are somewhat higher than the yields by the ethanolysis method, they are still not sufficiently high to be considered as representative building units. Indeed it seems more likely that these compounds result from the more easily cleaved portion of the lignin. In all probability (although not proved) this is the same portion that gives rise to the derivatives obtained by the other degradation procedures.

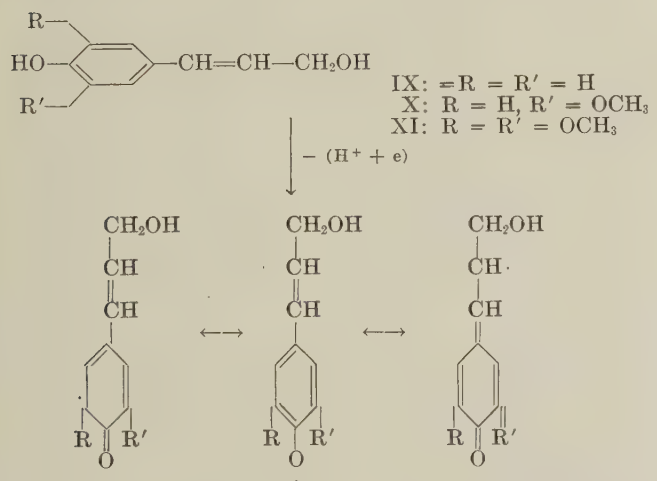
C. THE BIOGENESIS OF LIGNIN

Considerable attention has been given during the past few years to the problem of lignin biogenesis. Present theories (*47*) of ultimate lignification involve the polymerization of unstable, dehydrogenated radicals of the semiquinone type derived from any or all of the alcohols, *p*-coumaryl (IX), coniferyl (X), and sinapyl (XI) by dehydrogenase enzyme systems. The resulting *p*-quinone methide intermediates may then be stabilized to yield the elements of lignin structure for which evidence has been obtained as described earlier.

The pathway by which the individual monomers (IX, X, or XI) are synthesized from carbon dioxide has yet to be firmly established. Considerable evidence is available which indicates that shikimic acid is in-

volved in the synthesis of the aromatic ring (*48*, *49*). For hardwoods any plausible theory must perforce account for the different types of building units as well as the mode of their combination in the lignin polymer. As a result of such biogenetic studies, Freudenberg and co-workers (*50*, *51*) have obtained interesting data in support of the belief that hardwoods lignin is a mixed polymer of guaiacyl and syringyl nuclei containing units. In the presence of a mushroom dehydrogenase enzyme system they showed that although coniferyl alcohol (X) readily gave rise to a ligninlike polymer, sinapyl alcohol (XI) alone gave little product, but an equimolar mixture of these two alcohols gave an 80% yield of an almost colorless mixed polymer. The analyses of this synthetic lignin compared favorably with those of cuproxam beech lignin, the former being only slightly lower in oxygen content. Similarly this enzyme was found to be incapable of condensing syringaresinol, a dehydrogenase dimer of sinapyl alcohol, to a polymer in the manner it can for pinoresinol, the analagous dimer of coniferyl alcohol.

Although it may be argued that such *in vitro* biogenetic studies do not really aid in the knowledge of naturally produced lignin, and that it must remain to *analyze* rather than to *synthesize* to prove structure, this type of research has other valuable aspects. Information is thus being accumulated whereby selective enzymatic reactions can be utilized in the more general field of synthetic chemistry. It may well happen that the proof of structure of yet to be isolated lignin derivatives, of the dimer, trimer, or higher molecular size, may well depend on their enzymatic syntheses. In the opposite sense, microbiological methods of degradation (not discussed in this review) may well prove to be of immense value if and when there are available such systems which can effect selective cleavage of repetitive linkages of the macro lignin molecule.



In attempting to assess this multitude of facts that has been assembled regarding hardwood lignins, one is reminded again of the inquisitive men of Indostan and the "elephantine" size of the problem. An elegant appraisal of the outstanding problems in lignin chemistry has been made by Erdtman (*52*). Many of these problems have been emphasized earlier in this review. To obtain even a qualitative picture of the chemical nature of angiosperm lignin, it is necessary to superimpose on the proposed structural units outlined by Adler (*53*) for a typical gymnosperm, those units with the syringyl nucleus and for certain species those units with a *p*-hydroxyphenyl nucleus as well. The absolute percentage of each of these types of nuclei in protolignin is unknown; their mode of combination, either separately or in copolymer is unknown, and there is no real proof that the whole lignin substance is of a C_6—C—C—C polymeric nature. Until the yields of identified degradation products approach those required for a total carbon and methoxyl recovery and until the isolation and characterization of a typically dimeric or higher polymeric fraction is achieved, the problem will remain far from solved. The encouraging suggestion, made earlier, that for a syringyl-nuclei containing polymer, the number of possible linkages is minimized, and the chance of characterization therefore easier must be re-examined in the light of Freudenberg's research on the copolymeric nature of hardwood lignin.

Present evidence suggests that, as part of the wood cell, the lignin complex is chemically stable and only as a result of its liberation does it become a chemically reactive substance. It may be that much of the knowledge of the fundamental chemical composition must result from a study of chemical changes and extrapolation rather than by absolute measurement. It may be true, too, that the lignin chemist in his enthusiasm to isolate and unravel the secrets of this product of nature may have treated it with anything but gentleness and so created a monster which he cannot master. The lignin problem is nothing but a creation of the lignin chemist!

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Studies on the Chemistry of Aspenwood

IV. Oxidation of Aspen Lignosulphonates with Alkaline Cupric and Silver Oxide

IRWIN A. PEARL and DONALD L. BEYER

Quantitative and qualitative results of the alkaline cupric oxide and alkaline silver oxide oxidations of ammonia base *Torulopsis utilis* fermented aspen spent sulphite liquor and aspen basic calcium lignosulphonate were obtained. Arbitrary reactant ratios based on total solids were employed in these studies, and experimental results demonstrated that operative conditions must have been different in the separate experiments. Most compounds found were to be expected. These were vanillin, syringaldehyde, vanillic acid, syringic acid, *p*-hydroxybenzoic acid, acetovanillone, and acetosyringone. In addition, ferulic acid and *p*-coumaric acid were obtained, sometimes in relatively high yields. This is the first recorded instance of the obtainment of lignin degradation compounds containing three-carbon side chains from lignin oxidation reactions.

In continuing our studies on the chemistry of aspenwood, aspenwood lignin, and aspenwood lignosulphonates we investigated the oxidation of these materials with alkaline silver and cupric oxides under representative conditions previously evolved for coniferous lignosulphonate materials (*1*, *2*). The present paper reports preliminary studies on the oxidation of commercial lignosulphonate materials of predominantly aspen origin and the analysis of oxidation mixtures by rapid procedures developed since our earlier studies.

These preliminary studies were made on two commercial aspen lignosulphonates. One product was a *Torulopsis utilis*-fermented ammonia-base spent sulphite liquor of aspen origin and the other was a basic calcium lignosulphonate produced from a spent sulphite liquor of 85% aspen and 15% softwood origin. Conditions and reactant ratios for these preliminary experiments were chosen arbitrarily from the conditions found to give optimum results on coniferous lignosulphonates (*1*, *2*). Because of the insecure status of the methoxyl content of aspen "lignin" and because of the similar status of the "lignin" content of aspen lignosulphonate fractions or materials (*3*), all reactant ratios were determined on the basis of solids in the lignosulphonate starting materials. Similarly, data obtained are also reported on the solids basis.

The alkaline oxidation mixtures were acidified and extracted continuously with ether. The ether extracts were chromatographed in the butanol-2% aqueous ammonia, 10:3:3 butanol-pyridine-water and 0.3 *N* H_2SO_3 developers at 20°C., and the chromatograms were examined under ultraviolet light and sprayed with the bis-diazotized benzidine, 2,4-dinitrophenylhydrazine, ferric chloride, and Mäule reagents. On the basis of these chromatograms and comparison with known compounds, the presence of vanillin, syringaldehyde, vanillic acid, syringic acid, *p*-hydroxybenzoic acid, *p*-hydroxybenzaldehyde, acetovanillone, acetosyringone, ferulic acid, and *p*-coumaric acid were noted. In addition, several other as yet unidentified products were present in all cases. When present, the above noted compounds were determined quantitatively by chromatographic and spectrophotometric procedures recently developed in connection with other work in our laboratory on the alkaline hydrolysis of representative hardwoods and reported in detail previously (*4*, *5*).

The presence of acetovanillone and acetosyringone in the oxidation reaction products presented a problem in

Irwin A. Pearl, Senior Research Associate and Chief, and Donald L. Beyer, Research Aide, Lignin Chemistry Group, The Institute of Paper Chemistry, Appleton, Wis.

the quantitative analysis of these mixtures. The 6:1:1 heptane-*n*-butyl ether-water developer employed for the quantitative estimation of *p*-hydroxybenzaldehyde, vanillin, and syringaldehyde (*5*, *6*) failed to separate vanillin from acetovanillone and syringaldehyde from acetosyringone. Thus, new procedures were necessary for the determination of acetovanillone and acetosyringone and for *p*-hydroxybenzaldehyde, vanillin, and syringaldehyde in the presence of acetovanillone and acetosyringone.

QUANTITATIVE PROCEDURE FOR ACETOVANILLONE AND ACETOSYRINGONE-CONTAINING MATERIALS

In accordance with the procedure of Lemon (*7*, *8*), described in detail previously, (*4*), ultraviolet absorption curves were made for acetovanillone and acetosyringone in alkaline ethanol solution, and absorption maxima noted. These spectra were essentially the same as those reported by Lemon except that the wavelength of maximum absorption noted for acetovanillone was 350 mu rather than the 348 mu reported by Lemon. Concentration-optical density curves were obtained, and the factors for converting optical densities to concentrations in milligrams per liter were determined and found to be 6.67 for acetovanillone at 350 mu and 8.16 for acetosyringone at 362 mu.

It should be noted that the 352 mu wavelength of maximum absorption of vanillin (*4*) is very near that of acetovanillone. Because all of our earlier studies on vanillin utilized the 352 mu wavelength, optical density-concentration curves were determined for acetovanillone at this wavelength. The relationship was found to be a straight line, and the conversion factor was 6.84. The same procedure was applied to acetosyringone at the 368 mu wavelength of maximum absorption for syringaldehyde (*4*), and in this case, too, the optical density-concentration relationship was found to be a straight line with a conversion factor of 8.30.

A number of mixtures of vanillin and acetovanillone and of syringaldehyde and acetosyringone with both varying and constant total concentration were prepared. Total absorption was determined at 352 mu for the vanillin-acetovanillone mixture and at 368 mu for the syringaldehyde-acetosyringone mixture. Employing each component in a mixture as a known, the other component was calculated by subtracting the absorption due to the known from the total absorption and converting the residual absorption to concentration from the conversion factor for the unknown component. (See Table I.)

Table I. Analysis of Alkaline Ethanol Solutions Containing the Mixtures Vanillin-Acetovanillone and Syringaldehyde-Acetosyringone

Actual composition, mg./l.		*Composition calcd. from spectral data, mg./l.*	
Vanillin	*Aceto-vanillone*	*Vanillin*	*Aceto-vanillone*
2.00	2.00	2.07	2.09
4.00	4.00	4.07	4.09
2.00	8.00	1.99	8.03
8.00	2.00	8.09	2.02
4.00	6.00	4.07	5.96
Syring-aldehyde	*Aceto-syringone*	*Syring-aldehyde*	*Aceto-syringone*
2.00	2.00	1.97	1.97
4.00	4.00	3.91	3.92
2.00	8.00	2.00	8.02
8.00	2.00	7.97	2.03
4.00	6.00	4.04	6.08

The data of Table I indicate that the optical densities or absorptions are additive for the two sets of compounds under consideration, and that the maximum error encountered was 4.5%; the average error was much less. Accordingly, this spectrophotometric procedure was applied to actual mixtures to determine whether the individual components of the mixtures could be determined in the presence of each other when both concentrations were known.

An attempt was made to effect complete separation of acetovanillone from vanillin and acetosyringone from syringaldehyde by the procedure of Newcombe and Reid (*9*) employing sodium bisulphite-sprayed papers for chromatography of aldehyde-containing mixtures. The heptane-butyl ether-water developer gave satisfactory separation of the individual components of mixtures containing the two aldehydes and two ketones, but the presence of the sodium bisulphite on the paper precluded any spectrophotometric analysis of the eluates. A modified procedure was tried in which the paper was sprayed with sodium bisulphite solution only to the point at which the aldehydes were developed, but not including the portion of the paper containing the developed ketones. It was soon found that the R_f value for the aldehydes on sodium bisulphite-sprayed papers was a function of the amount of sodium bisulphite on the paper, and this value could not be maintained constant by the spraying technique. A satisfactory procedure was evolved for reproducible separations of these aldehydes and ketones under conditions which gave the same R_f value for the individual compounds.

METHOD FOR ALDEHYDE AND KETONE CHROMATOGRAPHY AND DETERMINATION

Whatman no. 1 paper, 61 cm. in length, was dipped in a 15% $NaHSO_3$ solution to a distance of 15 cm. from the top of the paper. This is only 7 cm. down from the baseline for chromatography. The paper was dried. The unknown extract was applied quantitatively as a streak along the baseline of the treated paper. In addition, a mixture containing known compounds was spotted alongside the streak. The paper was developed at 20°C. in the 6:1:1 heptane-*n*-butyl ether-water system. After development, the test strip was removed and sprayed with 2,4-dinitrophenylhydrazine reagent. In all cases, the aldehydes were held back by the bisulphite and found within the 7 cm. distance from the baseline. The acetosyringone moved approximately 10 to 15 cm. from the baseline, and the acetovanillone moved 30 to 35 cm. from the baseline in a development time of approximately 15 hr. R_f values were not recorded because the solvent front moved off the paper during the development time employed. The bands containing the desired materials were cut from the chromatograms, eluted with ethanol, made basic with potassium hydroxide, and processed in accordance with the general procedure for spectrophotometric analysis described in detail (*4*). The optical densities of the alkaline ethanol solutions were determined at the wavelength of maximum absorption noted earlier for the particular compound. The optical densities were converted to the concentration in milligrams per liter by the appropriate conversion factors. Actual weights were computed from this concentration value. In all spectrophotometric measurements, blanks were run on the dipped paper alone. A paper identical with that employed for

Table II. Cupric Oxide Oxidation of Aspen Fermented Spent Sulphite Liquor

Compound	Yield, % Ether extractives basis	Yield, % Original solids basis
Total ether extractives	...	22.9
Vanillin	13.8	3.16
Syringaldehyde	17.7	4.05
p-Hydroxybenzaldehyde	0.88	0.20
Acetovanillone	3.59	0.82
Acetosyringone	7.80	1.79
Vanillic acid	6.70	1.53
Syringic acid	7.67	1.75
p-Hydroxybenzoic acid	3.98	0.91

the determination was developed, cut, eluted, and processed along with and in the same manner as the streaked paper. In this manner, errors due to possible slight elution of salt were obviated.

The above procedure yielded the values for acetovanillone and acetosyringone. The original unknown mixtures were again streaked in the same manner on untreated papers and developed in the same heptane-butyl ether-water system. The bands containing mixed vanillin-acetovanillone and mixed syringaldehyde-acetosyringone were cut and processed as above. In this instance, the spectrophotometric readings were made at the wavelengths of maximum absorption of the aldehydes as noted above. Absorption due to the known amount of acetovanillone or acetosyringone was subtracted from the total absorption, and the remaining absorption was converted to the vanillin or syringaldehyde concentration. Analysis of a series of mixtures of all four compounds gave the following maximum errors: vanillin, 2%; syringaldehyde, 2.5%; acetovanillone, 1%; and acetosyringone, 2.5%.

EXPERIMENTAL PROCEDURE

Starting Materials

An ammonia-base *Torulopsis utilis* commercial spent sulphite liquor of peeled aspenwood (mixed *Populus tremuloides, P. grandidentata,* and *P. tacamahaca*) was obtained in November of 1956 from the Charmin Paper Products Co. of Green Bay, Wis. The liquor was concentrated in a circulating evaporator under reduced pressure to approximately one fourth of its original volume. The resulting concentrated liquor contained 31.8 g. solids per 100 g. of liquor, and the solids contained 10.76% methoxyl and 1.8% total sugars.

A sample of commercial aspen basic calcium lignosulphonate was obtained in May of 1957 from Marathon, Div. of American Can Co. of Rothschild, Wis. The spent sulphite liquor employed for the preparation of this material was the effluent from a calcium-base cook of approximately 85% aspen and 15% softwoods. The moisture content of this product was 8.03%, and the methoxyl content on the oven-dry basis was 9.74%.

Table III. Silver Oxide Oxidation of Fermented Aspen Spent Sulphite Liquor

Compound	Yield, % Ether extractives basis	Yield, % Original solids basis
Total ether extractives	...	7.34
Vanillin	10.6	0.60
Syringaldehyde	4.94	0.28
p-Hydroxybenzaldehyde	1.27	0.07
Acetovanillone	1.50	0.09
Acetosyringone	3.74	0.21
Vanillic acid	11.4	0.65
Syringic acid	13.5	0.77
p-Hydroxybenzoic acid	14.3	0.82
Ferulic acid	4.10	0.23

Oxidation of Fermented Aspen Spent Sulphite Liquor with Alkaline Cupric Oxide

A mixture of 460 g. of concentrated fermented aspen spent liquor containing 146 g. of solids, 450 g. of cupric hydroxide, 160 g. of C.P. NaOH, and 2200 g. of water was stirred and heated at 170°C. for 2 hr. in the 1-gal. stainless steel digester and processed as described earlier (*2*). The filtered alkaline reaction mixture was acidified with dilute sulphuric acid and extracted continuously in an air-agitated liquid-liquid extractor (*10*). The ether extract was evaporated under reduced pressure to yield 33.44 g. of ether extractives corresponding to 22.9% of the starting solids.

The ether extract was chromatographed qualitatively and quantitatively as described in detail in earlier papers (*4, 5*) and as modified by the acetovanillone and acetosyringone procedures described above. Quantitative results are given in Table II.

Oxidation of Fermented Aspen Spent Sulphite Liquor with Alkaline Silver Oxide

The procedure employed was a modification of that described in detail in earlier studies (*1*). Silver oxide, freshly prepared from 411 g. of $AgNO_3$ and one equivalent of NaOH, was placed in a 5-l. three-neck flask equipped with a reflux condenser, thermometer reaching below the ultimate liquid level, and a silicone oil-sealed stirrer. The flask was placed in a hemispherical heating mantle. With stirring, the silver oxide was covered with 1000 g. of water and treated with 367 g. of C.P. NaOH pellets. The temperature rose to 85°C. Stirring was continued, and when the temperature dropped to 66°C., 483 g. of concentrated fermented aspen spent liquor containing 154 g. of solids were added at one time. A vigorous reaction took place a few seconds after addition, and the temperature rose to 93°C. After reaction subsided, the mixture was heated to boiling with stirring under reflux for 4 hr. and allowed to cool with stirring. The cool mixture was filtered, and the metallic silver residue was washed thoroughly with water. The combined filtrate and washings were acidified with dilute sulphuric acid and extracted with ether in the air-agitated liquid-liquid extractor. The extract was processed as in the case of the alkaline cupric oxide oxidation. Results are given in Table III.

Oxidation of Aspen Basic Calcium Lignosulphonate with Alkaline Cupric Oxide

This experiment was performed in the same manner as the oxidation of the concentrated fermented aspen spent liquor except that 146 g. of aspen basic calcium lignosulphonate solids and 2500 g. of water were employed in place of 460 g. of spent liquor and 2000 g. of water. Data for this experiment are found in Table IV. In addition to the compounds shown in Table IV, qualitative chromatography indicated two new as yet unidentified compounds. The ferric chloride spray produced a strong blue spot with butanol-aqueous ammonia R_f 0.02 and butanol-pyridine-water R_f 0.05 and a strong purple spot with R_f's of 0.38 and 0.56 in the same two developers, respectively. Both of these compounds are still unknown.

Table IV. Cupric Oxide Oxidation of Aspen Basic Calcium Lignosulphonate

Compound	Yield, % Ether extractives basis	Yield, % Original solids basis
Total ether extractives	...	19.8
Vanillin	13.6	2.70
Syringaldehyde	11.6	2.31
p-Hydroxybenzaldehyde	1.00	0.20
Acetovanillone	2.79	0 55
Acetosyringone	5.54	1.10
Vanillic acid	4.04	0.80
Syringic acid	9.37	1.86
p-Hydroxybenzoic acid	0.58	0.12
Ferulic acid	4.75	0.94
p-Coumaric acid	0.62	0.12

Oxidation of Aspen Basic Calcium Lignosulphonate with Alkaline Silver Oxide

This experiment was performed in the same manner as the oxidation of the concentrated fermented aspen spent liquor except that 146 g. of aspen basic calcium lignosulphonate solids were added at one time as a dry powder to the warm mixture of silver oxide, alkali, and 2500 ml. of water. In this experiment no vigorous reaction took place immediately. Instead, reaction took place slowly, and the temperature gradually rose to 78°C. over 15 min. Suddenly, a vigorous reaction took place raising the temperature to 93°C. and depositing metallic silver in the reaction mixture. The quantitative data for this experiment are given in Table V. Qualitative chromatography indicated no aldehydes or ketones whatsoever. Several compounds never found in the past, however, were found. Compounds giving bright yellow spots with the Mäule spray reagents were found at R_f's 0.08 (0.30), 0.61 (0.81), and 0.65 (0.58) in the butanol-2% aqueous ammonia developer (values in parentheses are corresponding R_f values for the butanol-pyridine-water developer). The R_f 0.65 (0.58) compound gave a visible yellow spot before spraying. Two compounds giving strong negative fluorescence under ultraviolet light appeared at R_f's 0.61 and 0.65 in the butanol-aqueous ammonia developer. Both compounds had the same R_f of 0.58 in the butanol-pyridine-water system.

DISCUSSION OF RESULTS

The cupric oxide oxidation of aspen fermented liquor gave a yield of 22.9% on the basis of the starting solids. It should be noted that if the previously accepted value of 20 to 21% methoxyl be assumed for aspen lignin, the yield of ether solubles in this experiment would be approximately 45% on the basis of aspen lignin. It is interesting to note from the data of Table II that the yield of *p*-hydroxybenzoic acid is not greater than that expected by alkaline hydrolysis of the original wood (*4*), again indicating that this acid is not a product of oxidation of *p*-hydroxybenzyl moieties in aspenwood, but a product of hydrolysis alone, and occurring *in situ* in the wood as the benzoate moiety.

The quantitative data of Table III on the silver oxide oxidation of fermented aspen liquor were startling in several respects. Firstly, ferulic acid had never been obtained in any type of oxidation of lignosulphonate material in the past, but more unusual, vanillin and syringaldehyde had never been recovered in more than trace amounts from alkaline reaction mixtures containing silver oxide or reduced silver metal obtained by reduction of silver oxide. Earlier experiments on coniferous lignosulphonates and on vanillin alone indicated that vanillin was unstable in alkaline solution in the presence of silver oxide or silver metal (*1*, *11–14*). However, other studies on the use of technical caustic in silver oxide oxidations of vanillin (*15*) indicated that impurities present in technical caustics would inhibit vanillin reactions. Therefore, it is entirely possible that impurities present in the fermented aspen spent sulphite liquor might inhibit the oxidation of vanillin and the other aldehydes, syringaldehyde and *p*-hydroxybenzaldehyde. Furthermore, it is also possible that oxidation of ferulic acid was also inhibited. These possibilities together with the fact that a large portion of the extractives became ether-insoluble indicate that this particular experiment did not follow the pattern of previous silver oxide oxidations. The *p*-hydroxybenzoic acid yield in this experiment again falls within the range of the total *p*-hydroxybenzoic acid found in aspenwoods by direct alkaline extraction.

Table V. Silver Oxide Oxidation of Aspen Basic Calcium Lignosulphonate

Compound	Yield, % Ether extractives basis	Yield, % Original solids basis
Total ether extractives	...	10.0
Vanillic acid	3.1	0.31
Syringic acid	3.8	0.38
p-Hydroxybenzoic acid	3.6	0.36

The data of Table IV on the cupric oxide oxidation of aspen basic calcium lignosulphonate are somewhat different from those of Table II on a similar oxidation of aspen fermented spent sulphite liquor. The syringaldehyde vanillin ratio is much lower in the basic calcium lignosulphonate oxidation. This is to be expected to a certain extent because the basic calcium lignosulphonate contained a small proportion of coniferous material. However, the much lower acetovanillone yield in the basic calcium lignosulphonate oxidation cannot be explained unless we assume that the pretreatment given the basic calcium lignosulphonate material changed or removed structures in the lignin complex responsible for acetovanillone in the oxidation product. Such a structure might be 1-(4-hydroxy-3-methoxyphenyl)-3-hydroxypropan-1-one, which according to Kratzl (*16*) yields acetovanillone and formaldehyde by a reverse aldol mechanism. The yield of acetosyringone was affected to a less extent by whatever pretreatment influenced the acetovanillone yield, and the acetosyringone acetovanillone ratio was essentially the same in both oxidation experiments. It should be noted that the fermented liquor was prepared from ammonia-base liquor, and the basic calcium lignosulphonate from calcium-base liquor.

The *p*-hydroxybenzoic acid yield in this basic calcium lignosulphonate oxidation was much lower than in any previous oxidation of aspen lignosulphonate material. This is true for two reasons. The small proportion of coniferous lignosulphonate material would be responsible for a slight decrease in *p*-hydroxybenzoic acid formation, but more important, the fractional precipitation of aspen spent liquor with lime (Howard process) employed for the preparation of the basic calcium lignosulphonate left most of the *p*-hydroxybenzoic acid-yielding material in solution. Under conditions of preparation,

all *p*-hydroxybenzoic acid present *per se* in the spent liquor would also remain in solution, and only the *p*-hydroxybenzoate groups still attached to high molecular weight lignosulphonate material would be precipitated by the lime.

Again, the high yield of ferulic acid and the appearance of *p*-coumaric acid in the products of oxidation were surprising since neither of these acids had been noted in oxidations of lignosulphonate materials in the past. It should be noted that even in these experiments the ferulic and *p*-coumaric acids were determined only by paper chromatography. Indentification of these acids was made on the basis of specific fluorescence under ultraviolet light, R_f values in a number of developing solvents, and specific color reactions (*17*), and ultraviolet absorption spectra.

The absence of spots for aldehydes and ketones in the paper chromatograms of the silver oxide oxidation of aspen basic calcium lignosulphonate was unusual in view of the fact that high acetovanillone and acetosyringone yields were obtained on cupric oxide oxidation of this same basic calcium lignosulphonate material and that these two ketones along with vanillin and syringaldehyde were formed on silver oxide oxidation of fermented aspen spent sulphite liquor. It is possible that the two-stage reaction noted in this experiment was responsible for the nonappearance of acetovanillone and acetosyringone in this case. The quantitative data of Table V for the only three identified compounds in this experiment indicate that only a small portion of the ether extractives are accounted for by these identified compounds. The *p*-hydroxybenzoic acid is again very low in the oxidation products, but it is a little higher than in the cupric oxide oxidation of the same aspen lignosulphonate.

The experiments reported in this study indicate that reactant ratios based on total solids must have given widely different oxidizing conditions for the two aspen lignosulphonate materials employed. Although the exact nature of aspen lignin is unknown, it is apparent that some means, probably on the basis of methoxyl values, must be employed for its calculation so that a lignin-oxidizing agent ratio can be calculated.

The two-stage silver oxide reaction is being investigated further, and it is hoped to relate the nature of the products obtained before and after the second vigorous reaction to the composition of aspen and hardwood lignins.

The finding by paper chromatographic techniques of the three-carbon side chain acids, ferulic and *p*-coumaric, in the lignin oxidation products of aspen lignosulphonate materials indicate that maybe ferulic acid was present in coniferous lignosulphonate oxidation products obtained in the past. This possibility is being investigated, and the results will be reported in forthcoming papers.

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The Distribution and Availability of Hardwoods in North America

D. V. LOVE

The factors which influence the availability of hardwood, namely (1) the land area devoted to hardwood growth; (2) the inventory of present volumes; (3) the rate of hardwood growth; (4) its rate of utilization and waste; and (5) the extent to which man's interference with nature has influenced the picture, are discussed quantitatively. It is observed that the present total cut of all pulpwood—hardwood and softwood—in North America is approximately equal to the present allowable cut of hardwood pulpwood which is not at present being utilized for any purpose.

Statistics concerning the area of land in hardwood timber and the inventory volumes, growth, and utilization of hardwoods appear to be an absolute necessity in any appreciation of the situation suggested by the topic. Accordingly, from various sources (which include unpublished data provided on request) a comprehensive picture of the present hardwood situation is indicated in Tables II and IV, inclusive. These data have been reduced to graphic form for easier interpretation and are reproduced in Figs. 2 to 5, inclusive.

Because of the great variety of conditions which prevail within United States and Canada it was evident that some regional breakdown would be necessary if the topic was to be discussed in sufficient detail to serve the purposes of the meeting. Accordingly, regional areas were outlined in which conditions were reasonably consistent. These regions are defined, in terms of the political units included, and are shown in Table I. A map of North America illustrating the

D. V. Love, Associate Professor of Forestry, Faculty of Forestry, University of Toronto, Toronto, Ont.

Table I. Regional Breakdown of North America

Region	*Political units included*
Mexico	Mexico
United States	
Northeast	New England (Me., N. H., Vt., Mass., Conn., R. I.)
	Middle Atlantic (N. Y., Penn., W. Va., Md., Del., N. J.)
North Central	Lake States (Minn., Wis., Mich.)
	Central States (Iowa, Mo., Ill., Ind., Ohio, Ky.)
South	South Atlantic (Va., N. C., S. C.)
	Southeast (Tenn., Miss., Ala., Ga., Fla.)
	West Gulf (Ark., La., East Tex.)
Plains	(N. Dak., S. Dak., Neb., Kan., Okla., Tex.)
West	Northern Rocky Mountain (Mont., Idaho, Wyo.)
	Southern Rocky Mountain (Nev., Utah, Col., Ariz., N. Mex.)
	Pacific Northwest (Wash., Oregon)
	California
Alaska	Alaska
Canada	
Maritimes	Newfoundland, N. B., N. S., P. E. I.
Quebec	Quebec
Ontario	Ontario
Prairies	Manitoba, Saskatchewan, Alberta
British Columbia	British Columbia
North	Yukon, N. W. Territories

same regional breakdown is shown in Fig. 1. It must be appreciated that even within regions there will be considerable variation in forest conditions but the pertinent factors, the proportion of area in hardwood timber, the principal species and growth will be reasonably uniform.

Further tables and diagrams have been set up indicating the details respecting land area classification (Table II and Fig. 2), inventory volumes of hardwood (Table III and Fig. 3), and hardwood growth and drain (Table IV and Figs. 4 and 5).

A preliminary examination of these tables and diagrams suggested that certain of the regions could be eliminated without further consideration. This has definite advantages since it provides time for a more

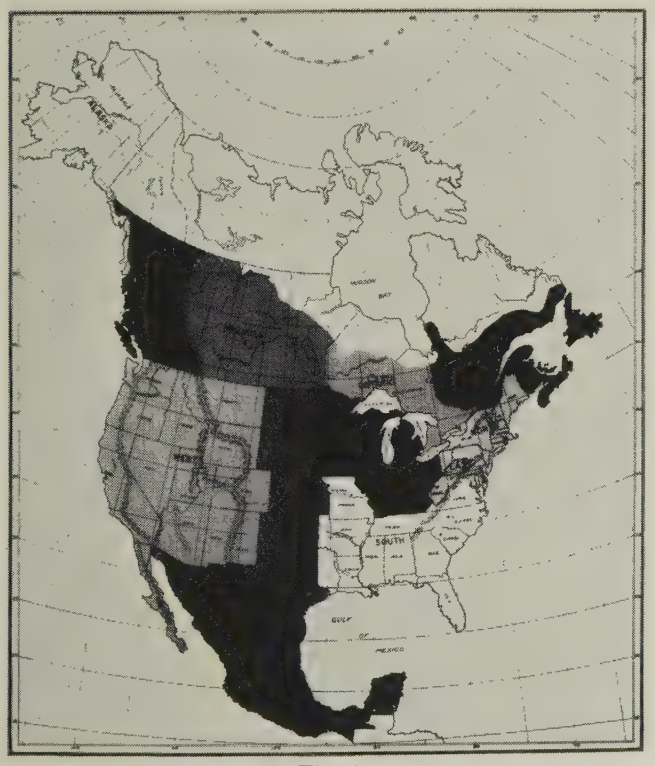

Fig. 1

Table II. Land Area Classification

Region	*Total land area, millions of acres*	*Commercial forest land* — *Millions of acres*	*Commercial forest land* — *Per cent of total area*	*Commercial hardwood forest*[a] — *Millions of acres*	*Commercial hardwood forest*[a] — *Per cent of commercial forest land*
Mexico[b]	487	49	10	33	67
United States[b]					
Northeast	127	73	58	54	74
North Central	314	96	31	81	84
South	329	193	59	112	58
Plains	373	5.5	1.5	4.8	87
West	760	117	15	4.0	3.4
Alaska	366	40	11	11	27
Total United States	2269	524	23	267	51
Canada[c]					
Maritimes	118	44	37	8	18
Quebec	335	139	41	18	13
Ontario	220	108	49	30	28
Prairie	441	128	29	34	26
British Columbia	230	79	34	6	8
North	933	49	5	5	10
Total Canada	2277	547	24	101	18
Total North America	5033	1120	22	401	36

[a] Forest land on which hardwood species predominate.
[b] Source "Timber Resources for America's Future," U.S.D.A. Forest Resource Report No. 14.
[c] Sources "The Outlook for the Canadian Forest Industries," Royal Commission on Canada's Economic Prospects, and Department of Northern Affairs and National Resources, Forestry Branch Bulletin 106.

detailed examination of those areas in which the possibility of establishment of hardwood pulp mills exists. From Table II it appears that in United States, the Plains, the West, and Alaska, and in Canada, British Columbia and the North could be eliminated without further ado. The basis for this action is simply that hardwood forest represents such a small proportion of the land that the concentrations of volume required for pulp manufacture would be very expensive to accumulate. It is quite possible, of course, that within these regions specific areas of concentration may occur but these will be rare and will not, in the near future, offer any real attraction to industry on anything but a limited scale. This decision may appear to lack the support of Table III which indicates inventory volumes for the regions in question of from 2.6 billion cu. ft for the Plains region to almost 20 billion cu. ft. for British Columbia. These inventory volumes are inclined to be somewhat misleading unless full account is taken of the fact that the volume in question is scat-

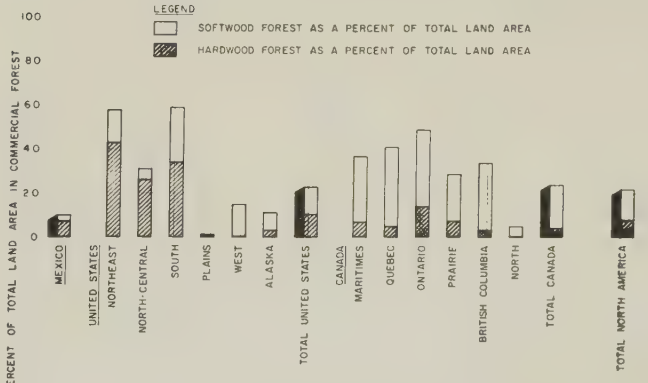

Fig. 2. Per cent of land area in hardwood forest (North America)

Table III. Inventory Volume (Net) of Hardwood Timber on Commercial Forest Land

Region	Sawtimber	Polewood	Total, millions of cu. ft.
	Millions of cu. ft.		
Mexico[a]	No breakdown possible		40,000
United States[a]	(25,000)	(15,000)	
Northeast	20,383	23,098	43,481
North Central	22,263	19,994	42,257
South	37,616	25,018	62,634
Plains	1,432	1,182	2,614
West	6,126	4,645	10,771
Alaska	No breakdown possible		8,854
	(5,000)	(3,854)	
Total United States & Alaska	87,820 (92,820)	73,937 (77,791)	170,611
Canada[b]			
Maritimes	4,688	2,529	7,217
Quebec	2,609	14,620	17,229
Ontario	15,692	14,535	30,227
Prairies	15,200	18,769	33,969
British Columbia	14,337	5,450	19,787
North	250	1,912	2,162
Total for Canada	52,776	57,815	110,591
Total for North America	170,596	150,606	321,202

[a] "Timber Resources for America's Future."
[b] "Bulletin 106," Department of Northern Affairs and National Resources, plus unpublished data provided on request.

tered over areas of from 230 million acres in British Columbia to 373 million acres in the Plains. Table IV generally bears out the decision to eliminate these areas since in all cases the limited growth available (maximum 222 million cu. ft. for B. C.) is largely dissipated in present utilization or in waste.

A further examination of Table IV reveals that Mexico is already utilizing hardwood in excess of production. This does not positively prove that a pulp mill could not find adequate stocks of hardwood in the tropical forests of Mexico but it does suggest that as the country develops its transportation systems the competition for wood will become severe. In comparison with many other areas in North America where hardwood growth is in excess of requirements Mexico is definitely not an attractive location for any large expansion of the hardwood pulp industry.

This leaves, as the areas most capable of supporting sizable expansion in the use of hardwood for pulp, Eastern United States—North and South, Eastern and Central Canada and the Prairie Provinces. Brief comments respecting these regions will be included in the following general statements concerning the factors of primary interest in this study.

The availability of any raw wood material depends upon these factors: (1) the area of land on which the timber in question exists or will grow, (2) the existing inventory of the timber, (3) the rate of growth of the timber, (4) the rate of utilization and waste under existing practices, and (5) the extent to which any or all of the above factors may be modified by man's interference with the normal processes of nature.

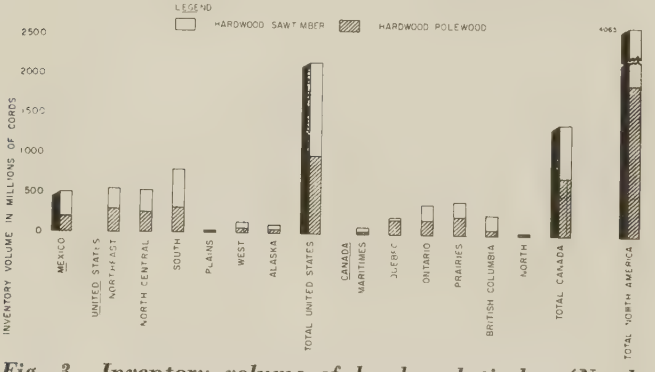

Fig. 3. Inventory volume of hardwood timber (North America)

These factors are to a great extent interrelated and in the time available it is not practical to attempt a comprehensive discussion of each factor. Certain points involving the influence of these factors on the availability of hardwood for pulp are pertinent to this discussion however and will be dealt with at this time.

LAND AREA AVAILABLE FOR HARDWOOD PRODUCTION

The land area available for hardwood production is not a static figure. Three factors of major importance tend to cause the land area available to hardwoods to change, often markedly. These are forest fires, logging operations, and tree planting.

It is dangerous to generalize respecting the behavior of forests after disturbance even in restricted areas since they often do not perform as predicted. In the larger area represented by this study lack of conformance would be guaranteed. However, by way of emphasizing the point in question, namely, that land area in hardwood is a variable with time it is pertinent to make a few observations that frequently are substantiated by performance on the ground. When mature uncut pine stands of Eastern Canada and United States are burned before a cut has been made these stands frequently regenerate to pine. However, if the fire occurs after the pine has been cut the area usually regenerates to hardwood. A cut-over area in which no fire has occurred usually produces hardwood also. Because of this, tremendous areas in the Eastern and Central United States which formerly supported pine now produce poplar and oak. In the southern states this trend toward an increase in the area of hardwood is also evident. To site a few examples, McCormack and Cruickshank (*1*) report that from 1936 to 1953 the hardwood stands of Georgia have increased from 22% of the forested area to 34%. Bryan (*2*) reports that in the Coastal Plain of Virginia the hardwood types have increased from 38% in 1940 to 45% in 1956, and Lockard (*3*) indicates that in the South "the hardwoods are truly ubiquitous." Numerous similar comments based on inventory and qualitative evaluation leave no doubt in the statement that, in general, the hardwood forests of the continent are, for one reason or another, steadily increasing.

While present-day cutting methods tend to increase the proportion of hardwood types, man in his desire to produce softwood is, in a small way, attempting to reverse this trend by planting softwood species especially pines and spruces. Although it receives a good deal of publicity planting is progressing at a very slow rate. By 1952 in the United States, 5,000,000 acres of successful plantation had been established and this represented 10% of the plantable area. In Canada the figure is known to be considerably less although no check of the statistics was made. Planting will not, for many years to come, appreciably alter the tendency of softwood forest types to shift to hardwoods on being cut over.

Table IV. Hardwood Growth and Drain

Region	Growth: Millions of cu. ft.	Growth: Rate as a % of inventory volume	Drain: Utilization, millions of cu. ft.	Drain: Waste,[b] millions of cu. ft.	Drain: Total millions of cu. ft.	Drain as % of growth
Mexico[c]	470	1.7	400	130	530	113
United States[c]						
Northeast	1,788	4.1	479	369	848	47
North Central	1,883	4.5	736	461	1,197	63
South	3,243	5.2	2,007	331	2,338	72
Plains	107	4.1	24	26	50	47
West	149	1.4	23	60	83	56
Alaska	88	1.0	Nil	0.5	0.5	0
Total United States	7,258	4.3	3,269	1,247	4,516	62
Canada						
Maritimes	197[e]	2.7	28[d]	8[e]	36	14
Quebec	381[e]	2.2	210[d]	16[e]	226	59
Ontario	667[d]	2.2	101[d]	123[d]	224	34
Prairies	888[d]	2.6	15[d]	139[d]	154	17
British Columbia	222[d]	1.1	8[d]	149[d]	157	70
North	101[e]	4.6	Unknown	...	...	0
Total Canada	2,456	2.2	362	435	797	32
Total North America	10,184	3.2	4,031	1,812	5,843	58

[a] For Canada the figures in this column are "allowable cut" which will exceed growth in those areas where mature timber predominates.
[b] Losses through fire, insects, and disease.
[c] Source, "Timber Resources for America's Future."
[d] Unpublished data provided on request.
[e] Source, "The Outlook for the Canadian Forest Industries."

HARDWOOD INVENTORY AND GROWTH

Inventory techniques are by no means uniform throughout North America and the resulting estimates vary considerably in accuracy. Accordingly, the data here presented should be considered as the best approximation available but still subject to considerable adjustment as more intensive surveys of volume and growth are undertaken.

Because of the trend toward an increase in the area of hardwoods previously noted and the failure generally to utilize the hardwood growth (as indicated in Table IV) the inventory of hardwoods is constantly increasing. The lack of accurate surveys of earlier dates precludes the possibility of a precise calculation for the Continent but published data (*4*) for eastern hardwoods in United States indicate a 20% increase in volume from 1944 to 1952. In the same period the increase in eastern softwoods was 16% and the decrease in western softwoods was 2%.

In spite of this tendency for the volume of hardwood to increase, much of the growth is accumulating on individuals or species of poor lumber or veneer quality.

These observations suggest that hardwoods for pulp are now and will continue for many years to be available in ever-increasing quantities in the eastern and northern regions of North America.

GROWTH AND ALLOWABLE CUT

Anyone who has had any contact with a practicing forester must surely have heard the term "allowable cut" and unless he has listened rather carefully to an explanation of the term he will probably have gained the impression that the allowable cut is equal to the growth of timber on the area in question. This impression is incorrect. Only in a perfectly regulated forest, of which there are virtually none in North America, is the allowable cut equal to the growth. In forests where an accumulation of unharvested growth exists the allowable cut exceeds the growth and in forests where overcutting has pertained the allowable cut is less than the growth.

In most regions of North America the growth of hardwood has not been harvested and hence has been allowed to accumulate. The allowable cut of this wood is therefore greater than the growth and exceeds the quantities shown in Table IV for United States (the data for Canada are the actual allowable cut).

The Eastern United States has a hardwood growth calculated as something in excess of 6.5 billion cu. ft. of which slightly more than 3 billion cu. ft. are utilized. One billion cubic feet are lost through fire or insect activity—a loss which would no doubt be reduced if a demand for the wood existed. The net growth available for expansion is therefore 2.5 billion cu. ft. or 32

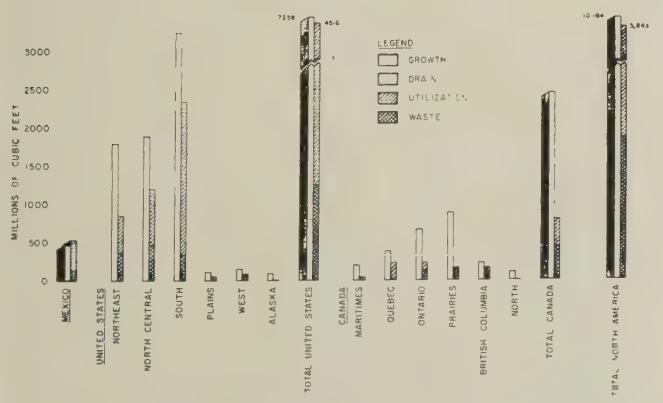

Fig. 4. Hardwood growth and drain (North America)

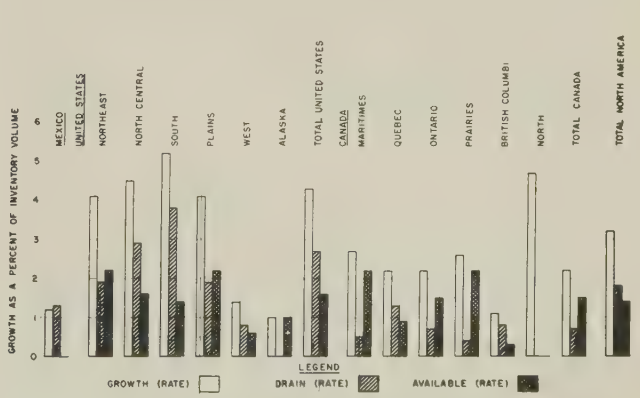

Fig. 5. Hardwood growth rate

Table V. Species Distribution in the Commercial Hardwood Regions

Region	Net available growth, millions of cu. ft.	Principle species groups in the inventory
United States		
Northeast	940	Beech-maple 30%, oaks 26%
North Central	686	Oaks 37%, poplar 12%, Beech-maple 11%
South	905	Oaks 40%, sweetgum 14%, blackgum 12%, hickory 9%
Total United States	2531	
Canada		
Maritimes	161	Poplar-birch 30%, Beech-maple 64%
Quebec	155	Popular-birch 67%, Beech-maple 28%
Ontario	443	Poplar-birch 76%, Beech-maple 18%
Prairies	734	Poplar 98%
Total Canada	1493	
Total North America	4024	

Source, "Timber Resources for America's Future." Bulletin 106, Department of Northern Affairs and National Resources plus unpublished data provided on request.

million cords per year and the allowable cut would exceed this by an appreciable but unknown amount.

In Canada the situation is similar but the figures given in Table IV are the calculated allowable cut and not the growth. The regions of Canada with which we are concerned have an allowable cut of 2.1 billion cu. ft. and a utilization of 0.3 billion cu. ft. with a further allowance of 0.3 billion cu. ft. for natural losses. The net growth available is therefore 1.5 billion cu. ft. or 19 million cords per year.

The surplus available allowable cut in hardwoods in North America exceeds 51 million cords. Compare this figure to the total cut of pulpwood, softwood and hardwood combined, in 1952 in the United States 35 million cords and in Canada 14 million cords. Thus, the present North American production of pulpwood of all species which equals 49 million cords is 2 million cords less than the allowable cut of hardwoods *which is not at present being utilized.* This is in excess of the quantity of wood available as leftovers from sawlog and sawmill operations of which there is a considerable amount.

SPECIES DISTRIBUTION IN THE COMMERCIAL HARDWOOD REGIONS

Many of the regions in which the hardwoods are found in quantity support a wide variety of hardwood species. It would be quite useless in a general discussion of this nature to attempt a detailed statistical record of the quantities of various species. Such records do exist for United States (*4*). For most of Canada the species arrangement is very simple as indicated in Table V.

Since species composition of the volume available is a matter of concern in pulp manufacture, a broad classification has been presented in Table V. It should be noted in reading this table that the principal species groups and the percentages associated therewith refer to the proportion of that species group in the *total* hardwood estimate for the region and not to the *net available growth.* It is perhaps misleading to combine net unused growth and the species proportions in the same table. However, because of the development of low quality trees in all species and in all regions and the need for partial cutting of small stems in stands of all species it is believed that the hardwoods available for pulpwood would in fact closely approximate the figures given in Table V.

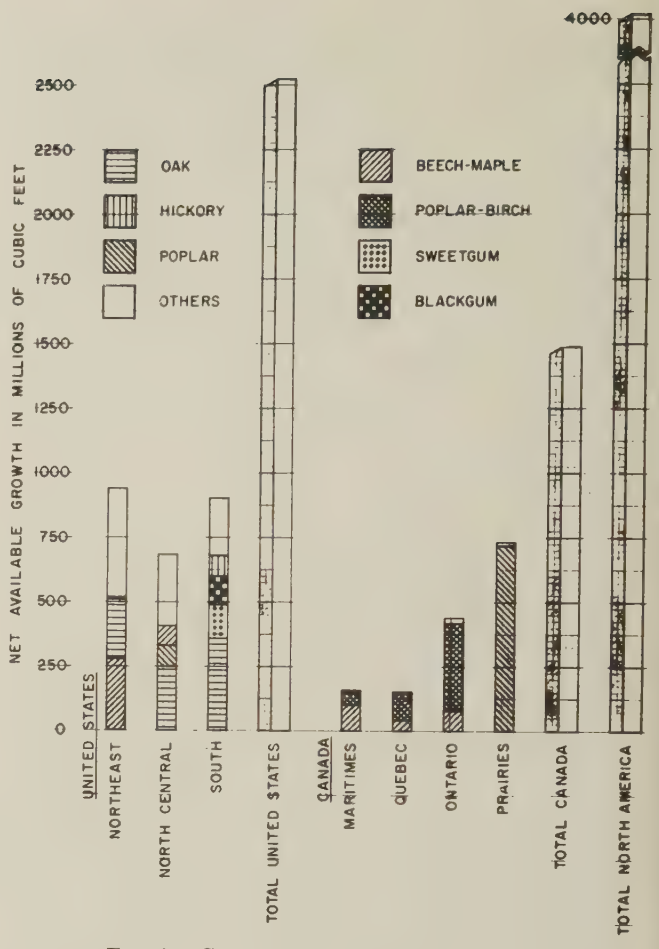

Fig. 6. Growth distribution by species

SUMMARY

It is believed that hardwoods can be cut commercially for pulp production in Eastern United States including the South, in Eastern and Central Canada and in the Prairie Provinces. The annual allowable cut of hardwood not presently utilized in these regions exceeds 51 million cords. In the United States the oaks are the most prevalent species followed by the beech and maple group which includes yellow birch. In Canada the principal species is poplar especially in the Prairie and Central regions; in the east the beech-maple group supersedes the poplar. White birch makes up a large part of the poplar-birch type in Eastern Canada.

REFERENCES

1. McCormack, J. F., and Cruickshank, James W., Forest Statistics for Georgia, 1951–53. U. S. Department of Agriculture, Forest Service.
2. Bryan, Mackay B., Forest Statistics for the Coastal Plain of Virginia. U.S.D.A. Forest Service.
3. Lockard, Charles R., Southern Hardwoods for Pulp and Paper, June, 1947. Issue of *Pulp and Paper Industry.*
4. Timber Resources for America's Future. U.S.D.A., Forest Resource Report No. 14.

Presented at the Third International Fundamental Research Conference sponsored by TAPPI and the Tech Section, CPPA, Montreal, Que., Sept. 15–17, 1958.

ASSOCIATION NEWS AND EVENTS

National Conferences

Fourth Deinking Conference, Roger Smith Hotel, Holyoke, Mass., Oct. 8–9, 1959.

Fourteenth Engineering Conference, Penn-Sheraton Hotel, Pittsburgh, Pa., Oct. 12–15, 1959.

Thirteenth Alkaline Pulping Conference, Robert Meyer Hotel, Jacksonville, Fla., Nov. 4–6, 1959.

Other 1959 Conferences

Oct. 1–3	Third International Conference of the Technical Association of the Paper and Printing Industries of Hungary, Budapest, Hungary.
Oct. 13–15	American Society for Testing Materials, Third Pacific Area National Meeting, Joint TAPPI-ASTM Committee on Petroleum Wax, Sheraton-Palace Hotel, San Francisco, Calif.
Oct. 26–28	Packaging Institute, Statler Hotel, New York, N. Y.
Nov. 9–10	National Paperboard Assoc., New York, N. Y.
Dec. 1–2	North American Pulp, Paper, and Paperboard Industrial Waste Conference, Edgewater Beach Hotel, Chicago, Ill.

Local Section Meetings

Pacific: Nov. 17, 1959, West Linn, Ore., "Coating and Printing"; Jan. 12, 1960, Longview, Wash., "Engineering"; April 15, 1960, Everett, Wash., Shibley Award Meeting; May 5–7, 1960, Bellingham, Wash., or Harrison Hot Springs, Annual Meeting; September, 1960, Salem, Ore., "Modernization."

Golden Gate District (Pacific Section): Oct. 6, 1959, Pittsburg, Calif. Plant visit to Dow Chemical Co., Dec. 1, 1959, "Economics of the Paper Industry"; Jan. 26, 1960, "Manufacturing and Marketing of Molded Paper Products"; March 29, 1960, "Recent Advances in Materials Handling"; May 17, 1960, R. M. True Memorial Lecture.

Lake States:

Delaware Valley: Oct. 22, 1959, Engineers Club, Philadelphia, Pa., "Felts"; Nov. 19, 1959, Engineers Club, Philadelphia, Pa., "Success in Safety"; Jan. 21, 1960, Engineers Club, Philadelphia, Pa., Operations Research Panel; Mar. 26, 1960, Benjamin Franklin Hotel, Philadelphia, Pa., 1960, Philadelphia Graphic Arts Conference; April 21, 1960, Engineers Club, Philadelphia, Pa., E. J. Albert Award Program; May 27, 1960, Beloit Eastern, Downingtown, Pa., tour and outing.

Kalamazoo Valley: Nov. 12, 1959, Inman's Restaurant, Galesburg, "Personal Development"; Dec. 3, 1959, Inman's Restaurant, Galesburg, "Coating"; Jan. 21, 1960, Harris Hotel, Kalamazoo, Papermakers' Get-Together; March 17, 1960, Inman's Restaurant, Galesburg, "Graphic Arts"; April 21, 1960, Inman's Restaurant, Galesburg, "Stock Preparation"; May 12, 1960, Harris Hotel, Kalamazoo, Recognition Night, WMU Awards; June 21, 1960, Annual Golf Outing, Gull Lake Country Club.

New England: Dec. 11, 1959, Past Chairmen's Night; April 8, 1960, Management Night; June 3–4, 1960, Chatham Bars Inn, Chatham, Mass., Spring Meeting.

New England (Eastern District): Armand's Restaurant, Route 9, Framingham, Mass.: Nov. 9, 1959; Jan. 11, 1960; March 14, 1960.

Ohio: Oct. 15, 1959, Manchester Hotel, "Food Additives," Speaker: A. A. Checchi; Nov. 10, 1959, Manchester Hotel, Middletown, Ohio, "Atomic Power Plant Development." This is a joint meeting with the Superintendents Association. Speaker: Ed Pandorf of C. G. & E. Dec. 10, 1959, Manchester Hotel, Middletown, Ohio, "Mechanics of Retention." Jan. 12, 1960, Manchester Hotel, Middletown, Ohio, "Clupak." Speaker: C. A. Shoudy. Feb. 11, 1960, Colony Restaurant, Swifton Center, Cincinnati, Ohio, plant tour through Formica, a division of American Cyanamid. March 15, 1960, Manchester Hotel, Middletown, Ohio, "Two-phase Flow in a Wet Web." Speaker: Joe Parker, Beloit Iron Works. April 14, 1960, Miami University, Oxford, Ohio, a tour of the new Paper School. Subject: "High Polymers." Speaker: Herman Mark. May 12, 1960, Hartwell Country Club, Cincinnati, Ohio, Ladies' night.

Ohio (Indiana District): Oct. 20, 1959, Visit to R. R. Donnelly Plant, Crawfordsville, Ind.; Nov. 17, 1959; Dec. 15, 1959; Jan. 19, 1960; Feb. 16, 1960; March 15, 1960; April 19, 1960; May 17, 1960; June 21, 1960, Golf outing.

Empire State:

Empire State (Metropolitan District): 465 Lexington Ave., New York, N. Y.

Empire State (Northern District): Woodruff Hotel, Watertown, N. Y.

Empire State (Central District): University Club, 431 E. Fayette St., Syracuse, N. Y.

Empire State (Western District): Prospect House, Niagara Falls, N. Y.

Empire State (Eastern District): Queensbury Hotel, Glens Falls, N. Y.

Maine-New Hampshire: October 23–24, 1959, Lafayette Hotel, Portland, Me., Fall Meeting—"Stock Cleaning" June 17–18, 1960, Mountain View House, Whitefield, N. H.; Annual Meeting.

Chicago: Chicago Bar Association, 29 S. LaSalle St., Chicago, Ill.

Chicago (St. Louis District): Le Chateau Restaurant, 10405 Clayton Road, St. Louis, Mo.,

Lake Erie: Oct. 15, 1959, Ohio Boxboard Co., Rittman, Ohio, plant tour; Nov. 19, 1959, Cleveland Engineering and Scientific Center, "Paper Requirements, Inks, Processing Advances and Problems of the Printing Industry"; Jan. 21, 1960, Cleveland Engineering and Scientific Center, Conference on Pulp and Paper Testing Equipment and Methods Including Laboratory Coating and Sheetmaking Techniques; Feb. 18, 1960, Colton Chemical Co., 6626 Union Ave., Cleveland, Ohio, plant tour; March 17, 1960, Cleveland Engineering and Scientific Center, "Off-Machine Coating with Emphasis on Waxing and Barrier Types and Base Paper Requirements."

Southeastern: Nov. 20–21, 1959, King and Prince Hotel, St. Simons Island, Ga.

Virginia-Carolina

Italian

Gulf Coast

Papermakers and Associates of Southern California

1960 Annual Meeting, Technical Section Canadian Pulp and Paper Association

The 1960 Annual Meeting of the Technical Section, Canadian Pulp and Paper Association, will take place at the Queen Elizabeth Hotel, Montreal, on the last Tuesday, Wednesday, Thursday, and Friday of January, the 26th, 27th, 28th, and 29th. Sessions will begin at 2:00 p.m. on Tuesday and will end at noon on Friday; the registration desk will open at 9:00 a.m. on Tuesday. A products display will be held concurrently with the meeting.

Requests for accommodation should be addressed to Nelson Verge, rooms division manager of the hotel. Give the date and time of your arrival, and tell the hotel if you are a member of the Technical Section. The Technical Section does *not* handle room reservations.

Anyone wishing to present a paper at this meeting should contact Douglas Jones, executive secretary, Technical Section, Canadian Pulp and Paper Association, 2280 Sun Life Building, Montreal 2, Que.

Report of the Nominating Committee

The 1960 TAPPI Nominating Committee consisting of Curt A. Young, Continental Can Co., New York, N. Y., chairman; E. O. Ericsson, Puget Sound Pulp & Timber Co., Bellingham, Wash.; R. C. Germanson, KVP Co., Kalamazoo, Mich.; James R. Lyon, Alton Box Board Co., Alton, Ill.; and Robert H. Simmons, Bureau of Engraving and Printing, Washington, D. C., have presented the following nominees for officers and executive committeemen of the Technical Association of the Pulp and Paper Industry to be voted on by mail ballot of the Association active and corporate members prior to the annual meeting of the Association in 1960:

President (1 year): James R. Lientz, Union Bag-Camp Paper Corp., Savannah, Ga.
Vice-President (1 year): Harold M. Annis, Oxford Paper Co., New York, N. Y.
Executive Committeemen (3 years):
John M. McEwen, Pulp Division, Weyerhaeuser Timber Co., Everett, Wash.
Alfred H. Nadelman, Western Michigan University, Kalamazoo, Mich.
Kenneth D. Running, Halifax Paper Co., Roanoke Rapids, N. C.
Horace A. Spencer, Knowlton Brothers, Inc., Watertown, N. Y.

Note: Article VII, Sec. 3, TAPPI Constitution: "Other nominations, if signed by at least 25 voting members, may be submitted to the Secretary of the Association, in writing, on or before November 20, and if each nomination so made is accepted in writing by the nominee, the name shall be placed on the official ballot."

Review of TAPPI Research Policy

At the February, 1959, meeting of the Executive Committee, Ward Harrison, former president of TAPPI, appointed a committee to review TAPPI research policy. The object of this work is the formulation of a realistic policy which will permit TAPPI to serve its individual members, and the industry, most effectively within the physical limitations of the organization.

The committee comprises John L. Parsons, consultant, of Rye, N. H., and chairman of the present Research Appropriations Committee; John H. Heuer, vice-president and manager of manufacturing, Great Northern Paper Co.; Frederic S. Klein, mill manager, Byron Weston Co.; and is headed by John R. Curtis, director of operations research for the Scott Paper Co.

In June the committee prepared a rather extensive questionnaire which was forwarded to division chairmen, local section chairmen, and to certain key individuals in the industry. The response to the questionnaire was most gratifying and many valuable comments, criticisms, and recommendations were received.

On July 30, 1959, the committee met in Boston to discuss the results and to draft recommendations and a statement of policy. The committee reported a most constructive meeting and feels that much has been done to clarify the current research policy problems. Their final report will be presented to the Executive Committee at its meeting in September, 1959. It is hoped that shortly thereafter the results will be transmitted to the membership. This publicity will probably appear in *Tappi* and at the February, 1960, meeting in New York.

Fifth Annual Conference—Study Group of European TAPPI Members

General Report

The conference which was held in Noordwijk, Netherlands, June, 4–6, 1959, was attended by 258 members and 54 ladies, a somewhat higher attendance than at the Fourth Conference in 1958 in Turin (254 and 40, respectively). The registrations were divided as follows:

Netherlands	68	Czechoslovakia	3
Great Britain	40	Denmark	3
Germany	38	United States	3
Italy	33	Egypt	2
France	21	Israel	2
Sweden	13	Poland	2
Switzerland	5	Belgium	1
Yugoslavia	5	Malaya	1
Finland	4	Morocco	1
Hungary	4	Norway	1
Spain	4	South Africa	1
Austria	3		

Thus, 23 countries were represented. This shows that the annual conference of the Study Group of European TAPPI Members has become a truly international gathering which attracts attendance even from countries outside Europe.

The conference was organized by a committee consisting of:

Prof. Ir. T. Tromp, Papierfabriek Gelderland, Nijmegen
Ir. G. van Nederveen, Fibre Institute T.N.O., Delft
Dr. F. M. Muller, Netherlands Experiment Station for the Utilization of Straw, Groningen

The committee was assisted, in the administration and registration of the Conference, by the Bureau of the Society for the Advancement of the Knowledge of Materials.

The conference of the Study Group was preceded by a symposium on straw pulp, organized by the same people, under the auspices of EuCePa (the Liaison Committee of the National Associations of pulp and paper technicians in Europe). The majority of members (191) attended both the EuCePa Symposium and the European TAPPI Conference. Both meetings were sponsored by the following organizations:

Society of Netherlands Paper Manufacturers, Haarlem
Society of Netherlands Strawboard Manufacturers, Groningen
Straw Pulp Mill Phoenix, Veendam
Cooperative Strawboard and Straw Pulp Mill Eendracht, Appingedam
Straw Pulp Mill Sove, Arnhem

The conference was held in the hotel "Huis ter Duin" at Noordwijk, where the registration office was located and where the majority of members were accommodated. This considerably facilitated the informal talks and exchange of opinions which form such an important part of an international meeting.

Technical Program

Wednesday, June 3

An excursion was arranged for Wednesday, June 3, which was open to members of both the EuCePa Symposium and the

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TAPPI Conference. Coaches brought the party in three groups to Arnhem where one group visited the Sove alkali-chlorine straw pulp mill in the morning and, in the afternoon, the Openair Museum where a seventeenth century paper mill could be seen in operation; a second group visited the Openair Museum in the morning and the Sove mill in the afternoon, while a third group, including the ladies, went to the Kröller Müller Museum in the morning and joined the first group at the Openair Museum in the afternoon. The first and second groups were the guests of the Sove mill at lunch, where Prof. Tromp thanked the Management for their cordial reception.

Karl Adamik, Technical University, Graz, retiring president

At night, the members of both meetings were the guests of the Society of Netherlands Paper Manufacturers at a cocktail party and buffet supper, where they were welcomed by the president of this society, H. F. de Graaf, who expressed his appreciation of the interest shown by members in the research on pulp and paper. The hospitality and congenial atmosphere at this party greatly contributed to the success of these meetings.

Thursday, June 4

On behalf of the organizing committee, Dr. Muller welcomed members to the conference. He explained the necessity for a small country such as the Netherlands to combine the organization of the TAPPI Conference with that of the EuCePa Symposium, which were both to be held in the same year; the large "overlap" in the attendance of these meetings had shown both the feasibility of this organization and the advantage to the participants of both meetings.

Prof. Adamik, President of the Study Group, then opened the conference, with a particular welcome to Philip E. Nethercut, Secretary-Treasurer of TAPPI.

The morning session, under the presidency of Prof. Adamik, continued with the following papers:

Dipl. Ing. O. Imset, Oslo—"The Laboratory Testing of Pulp in Relation to Paper Properties"
Prof. Dr. F. Wultsch, Graz—"New Results in the Production of Hardwood and in Their Use for Various Grades of Paper"

In the afternoon session, Dr. H. F. Rance, presiding, the following papers were read:

Dr. T. H. Frankel, London—"Manufacture of Bleached Hardwood Pulp by the Neutral Sulphite Process"
J. Arguillere, St. Etienne de Rouvray—"Paper Pulp from Scrubwood"
Prof. G. Vamos, Budapest—"Evaluation of Straw Pulp and Hardwood Pulp Mixtures"

Friday, June 5

The technical program continued with the following papers. Morning session, Dr. F. M. Muller presiding:

Dr. Ing. M. Bonavia, Lugo di Vicenza—"The Pomilio Process for 100% Straw Pulps"
Dr. J. Grant, London—"Printability Problems of Short-Fibered Papers with Special Reference to Straw"
Dr. U. Poggianti and Dr. G. Moltedo, Chieti/Rome—"Offset Papers Containing High Straw Pulp Proportions"
Dr. G. Botto Micca and Dr. E. Grandis, Turin—"Effects of Various Percentages of Monosulphite Straw Pulps for Packing Papers"

Afternoon session, Dr. G. Raimondo presiding:

Dr. A. H. H. van Royen, Delft—"Straw Pulp in Paper"

F. M. Muller, Netherlands Experiment Station for the Utilization of Straw, president

I. F. Hendry, Wiggins Teape & Co., Ltd., secretary-treasurer

D. Borruso and G. Ceragioli, Milan—"Effect of Poplar Mechano-Chemical Pulps on Wet and Dry Properties of Various Newsprint Furnishes"
Dr. P. Colombo, Dr. D. Corbetta, A. Pirotta, G. Ruffini, and A. Sartori, Milan—"The Vita Mayer and Co. Plant for Continuous Production of Poplar Cold Soda Pulp: Results of One Year Operation and Pulp Properties"
Dr. D. Corbetta, Dr. P. Colombo, A. Pirotta, G. Ruffini, and A. Sartori, Milan—"Laboratory Studies with the Vita Mayer Process on Pulpmaking Properties of Some Hardwood Species"

At the end of the afternoon session the general meeting was held, the minutes of which are given at the end of the report.

Saturday, June 6

This day was devoted to an excursion to Groningen. Members were conveyed by coach from Noordwijk to Leiden Station, and from there by special train to Groningen. From Groningen they were taken by coach to City Park, where they lunched as guests of the mills they were to visit during the afternoon. After lunch, Dr. Muller expressed the gratitude of members for the hospitality extended to them in Groningen. The party was then split into five groups to visit the following organizations, respectively:

Scholten's Board and Paper Mill, Sappemeer
Straw Pulp Mill Phoenix, Veendam
Cooperative Strawboard and Straw Pulp Mill Eendracht, Appingedam
Netherlands Experiment Station for the Utilization of Straw, Groningen
Ladies' trip to Menkemaborg, Uithuizen.

Members were brought back to Groningen station whence the party returned by train to Leiden and from there by coach to Noordwijk.

LADIES' PROGRAM

As well as the special visits for the ladies included in the excursions to Arnhem and Groningen, a ladies' trip was organized by The Hague Hospitality Committee on Thursday, June 4, to Amsterdam, where a boat tour through the canals was made and a visit paid to the Rijksmuseum (picture gallery).

ANNUAL GENERAL MEETING

Due to the unavoidable absence of the Secretary, no minutes of the previous meeting were read and no Secretary's report was presented.

Election of Officers

On account of the frequent absence of Dr. Schönemann, some German members had expressed the wish to nominate another German representative to the Committee. After discussion among German members present, Dr. Ing. K.

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FEBRUARY 8 MONDAY

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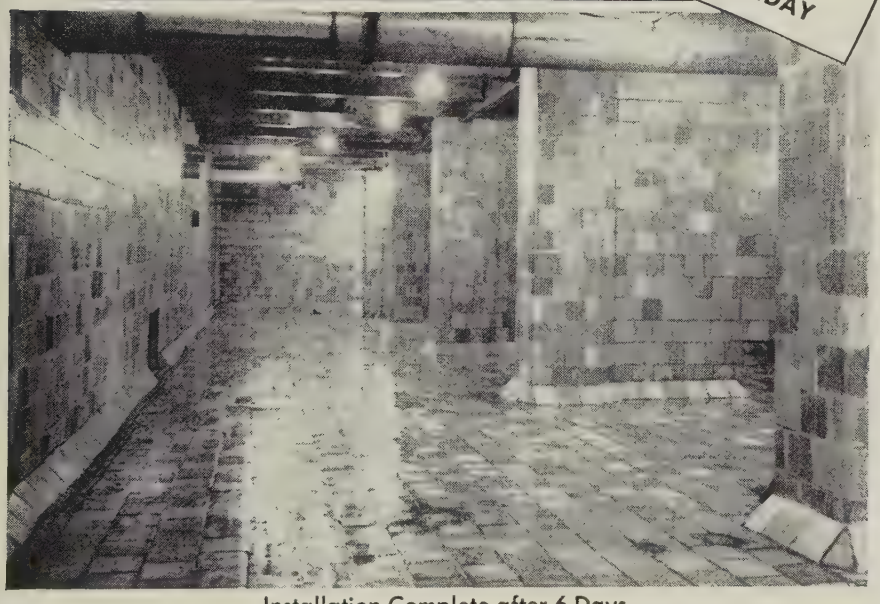

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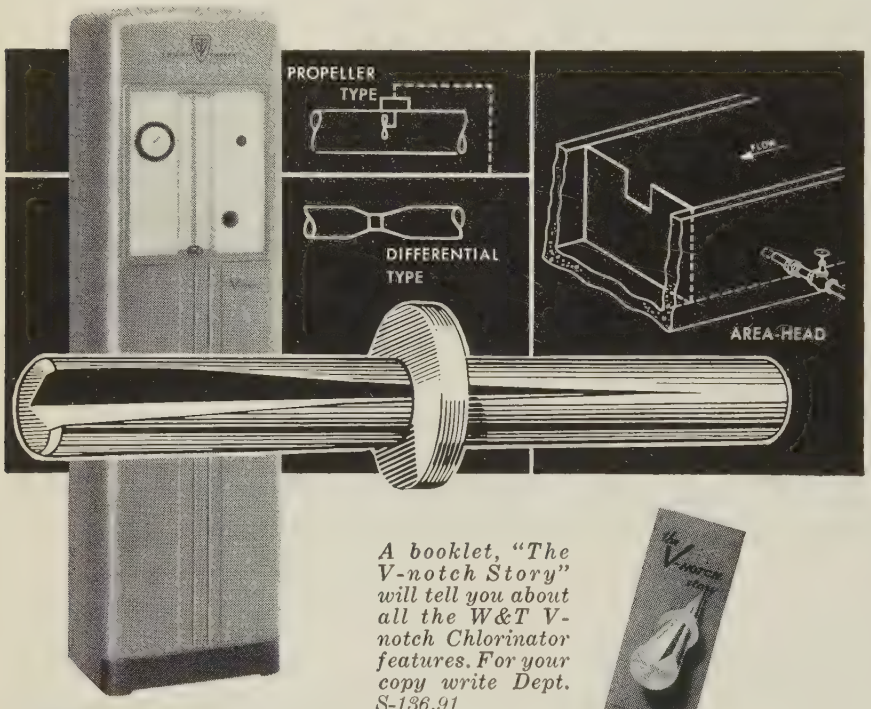

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Sieber, Alfeld/Leine, was nominated and elected to serve on the Committee.

Prof. Adamik tendered his resignation from the office of President, and Dr. F. M. Muller was nominated as his successor; Prof. Adamik was nominated as a Vice-President. These nominations were received with acclamation and carried.

No further nominations being received, the Committee now consists of: President: Dr. F. M. Muller; Vice-Presidents: Dr. P. Bersano and Prof. K. Adamik; Secretary/Treasurer: Mr. I. F. Hendry, Mr. P. Ceuterick of Belgium, Mme. H. Fourcaut of France, Dr. K. Sieber of Germany, Dr. H. F. Rance of Great Britain, Mr. C. G. von Essen of Sweden, Mr. R. Naville of Switzerland, and Prof. G. Vamos of Hungary.

Future Program

At the General Meeting in September, 1958, in Turin, mention was made of the possibility of organizing the 1960 Conference on a boat off the Baltic coast of Sweden. As the Committee had recently received information that it would be difficult to carry out this plan in 1960, a different country had to be sought. Due to the absence of some of its members, the Committee had not been able to work out a definite proposal for next year's Conference, and asked for the authorization of the meeting to take a decision on this matter as soon as possible. This proposal was carried by the meeting.

ANY OTHER BUSINESS

One of the objections brought forward against the TAPPI Study Group was that nonmembers of TAPPI had no access to the Annual Conference. Although the organizing committees had always taken a liberal stand in this matter, it was proposed that nonmembers who wished to be informed of annual conferences should give their names to the secretary so that they could be inscribed in a register of "affiliated" members without voting rights. This proposal was approved by the meeting.

Concerning relations with EuCePa, it was stated that these had improved, although some opposition still existed. It was proposed that those members of the Committee of the Study Group who would be in Paris at the occasion of the EuCePa Symposium on Printability next fall should take this opportunity to discuss these relations with members of the EuCePa Committee. This proposal received the approval of the meeting.

F. M. MULLER, *President*

PERSONAL MENTION

Items About New and Old Tappimen

New TAPPI Members

Alfred W. Anish, Chemist, Crane & Co., Dalton, Mass., a 1934 graduate of Clark University.

Seymour S. Beneroff, Sales Manager, President Container, Inc., Brooklyn, N. Y., a 1953 graduate of the College of the City of New York.

Edgar S. Borup, Director, Amerika-Haus Vienna, U. S. Information Service, Vienna, Austria.

Claud L. Brown, Silviculturist, Texas Forest Service, College Station, Tex., a 1958 graduate of Harvard University with a Ph.D. degree.

Charles C. Bush, Power Plant Superintendent, Union Bag-Camp Paper Corp., Savannah, Ga., a 1930 graduate of Alabama Polytechnic Institute, Auburn, Ala.

Luis Bustamante E., Jefe de la Seccion de Celulosas, Instituto Forestal de Investigaciones y Experiencias, Madrid, Spain, a 1945 graduate of Escuela Especial de Ingenieros de Montes.

Chih-Yao Chu, Chief Engineer, Pao Feng Industrial Co. Ltd., Taipei, Taiwan, China, a 1943 graduate of Chao-Tung University.

Samuel D. Coleman, Chemist, Polymer Industries, Springdale, Conn. Attended the Massachusetts Institute of Technology.

Jerold R. Dearwater, Technical Service Engineer, The Champion Paper & Fibre Co., Hamilton, Ohio, a 1952 graduate of the University of Cincinnati.

Franklin M. deBeers, Jr., Manager, Special Finishes Dept., Enterprise Paint Mfg. Co., Chicago, Ill., a 1934 graduate of Cornell University.

Johannes B. De Bie, Assistant Manager, Dorr-Oliver N. V., Amsterdam, Holland, a 1931 graduate of the Technical University.

Frank A. DiGioia, Chief Chemist, General Latex & Chemical Corp. of Georgia, Dalton, Ga., a 1946 graduate of Harvard University.

Samuel T. Douglass, Jr., Salesman, Shawinigan Resins Corp., New York, N. Y., a 1935 graduate of the Stockbridge School of Agriculture.

Horacio R. Farias, Papermaking Superintendent, La-Papelera Argentina S.A., Bernal, Argentina.

David A. Finnegan, Chief Engineer, Arkwright Finishing Co., Fiskeville, R. I., a 1948 graduate of the Massachusetts Institute of Technology.

Philip H. Germann, Student, Allied Paper Corp., Kalamazoo, Mich., a 1959 graduate of Western Michigan University.

James S. M. Gourlay, Chief Chemist, R. Collie & Co. Pty Ltd., North Melbourne, Australia, a 1952 graduate of the Royal Melbourne Technical College.

Mauri T. Happonen, Paper Mill Superintendent, United Paper Mills Ltd., Valkeakoski, Finland, a 1952 graduate of the Technical University of Finland.

Jacquelin E. Harvey, Jr., Research Director, Harvey-Reynolds, Inc., Cambridge, Md., a 1919 graduate of the University of Missouri.

Mistuo Hirata, Chief, Technical Dept., Honshu Paper Mfg. Co. Ltd., Fuji-shi, Shizuika-ken, Japan, a 1925 graduate of Kiryu Technical College.

William Holtzman, Project Engineer, Hudson Pulp & Paper Corp., Palatka, Fla., a 1959 graduate of The Institute of Paper Chemistry with a Ph.D. degree.

Ralph L. Horst, Jr., Engineer, Aluminum Co. of America, New Kensington, Pa., a 1948 graduate of Columbia University.

K. A. Isaac, Librarian, Forest Research Institute and Colleges, New Forest, Dehra Dun, India, a 1953 graduate of the University of Delhi.

William M. Jarnagin, Director of Sales Development, Krause Milling Co., Milwaukee, Wis., a 1945 graduate of the University of Tennessee.

Birger E. Jensen, Managing Director, A/S Greaker Cellulosefabrik, Greaker, Norway.

Richard H. Jones, Research Engineer, Champion Paper & Fibre Co., Hamilton, Ohio, a 1959 graduate of Western Michigan University.

Duane R. Keck, Research Engineer, Champion Paper & Fibre Co., Hamilton, Ohio, a 1958 graduate of Purdue University.

Myron W. Kiebler, Jr., Manager, The Glidden Co., Southern Chemical Div., Port St. Joe, Fla., a 1937 graduate of Carnegie Institute of Technology.

Armando B. Lorenzon, Pulp Mill Superintendent, La Papelera Argentina S.A., Bernal, Argentina.

Harry B. Lowell, Market Development, Dewey & Almy Chemical Div., W. R. Grade & Co., Cambridge, Mass., 1951 graduate of Massachusetts Institute of Technology.

Ralph P. Mahoney, Research Engineer, Beloit Iron Works, Beloit, Wis., a 1951 graduate of the University of Maine.

Lynn C. Mapes, Assistant Production Manager, *The Reader's Digest*, Pleasantville, N. Y., a 1953 graduate of Northwestern University.

Roger W. Martin, Research Manager, Marion, Son & Hall, Ltd., Bristol, England, a 1939 graduate of Liverpool University.

Henry H. Nichols, Engineer, Box Technical Dept., Union Bag-Camp Paper Corp., Savannah, Ga., a 1954 graduate of the University of Florida.

Takenao Noda, Director Chief, Production Dept., Honshu Paper Mfg. Co. Ltd., Chuo-ku, Tokyo, Japan, a 1922 graduate of Yonezawa Technical College.

James L. Olmstead, Market Research Analyst, Hooker Chemical Corp., Niagara Falls, N. Y., a 1954 graduate of the University of Buffalo.

Andre J. Pelletier, Research Chemist, The Mead Corp., Chillicothe, Ohio, a 1955 graduate of Lowell Technological Institute.

Clyde S. Reynolds, Vice-President and General Manager, Thurso Pulp & Paper Co., Thurso, Que., Canada, a 1940 graduate of Purdue University.

Henry Rinderknecht, President, Gessellschaft fur Holzstoffbereitung, Basle, Switzerland.

Robert G. Rohwer, Senior Chemist, Grain Processing Corp., Muscatine, Iowa, a 1943 graduate of the University of Nebraska.

Davis, M. Schroeder, Sales and Service, Central Soya Co., Inc., Chicago, Ill., a 1949 graduate of Michigan State College.

Gerald Skidmore, Technical Sales Representative, Archer-Daniels-Midland Co., Cincinnati, Ohio, a 1954 graduate of Purdue University.

William H. Somers, Coatings Development Chemist,

Dow Chemical Co., Midland, Mich., a 1957 graduate of Western Michigan University.

Kikuo Sugai, Planning Engineer, Takasage Netsugaku Kogyo K. K., Chiyoda-ku, Tokyo, Japan, a 1951 graduate of Tokyo Institute of Technology.

Minoru Sumi, Sales Engineer, F. Kanematsu & Co. Ltd., Chiyoda-ku Tokyo, Japan, a 1950 graduate of Sophia University.

Cesare Toniolo-Augier, General Manager, Enrico Toniolo & Figlio, Milano, Italy.

Glenn W. Torres, Coating Superintendent, International Paper Co., Moss Point, Miss., a 1948 graduate of the University of Alabama.

Harold V. Townsend, Manager, Martin Paper Products Ltd., New Westminster, B. C., Canada.

Lambert VanHaren, Director of Research and Development, Solvay et Cie, Brussels, Belgium, a 1933 graduate of Liege University.

Lawrence L. Wallace, Assistant Chief Civil Engineer, John G. Hoad & Associates, Inc., Ypsilanti, Mich., a 1950 graduate of the University of Detroit.

Frederick F. Wangaard, Professor, Yale University, School of Forestry, New Haven, Conn., a 1939 graduate of the New York State College of Forestry with a Ph.D. degree.

Leonard L. Wolfson, Head Microbiologist, Nalco Chemical Co., Chicago, Ill., a 1951 graduate of the University of Chicago.

James F. Zavatone, Quality Control Manager, Container Corp. of America, Medford, Mass., a 1954 graduate of the University of Notre Dame.

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TAPPI Notes

J. Marks Abernathy of the American Cyanamid Co. has been transferred from Kalamazoo, Mich., to Mobile, Ala., as Plant Superintendent.

Robert F. Avery, formerly of the Koppers Co. is now a Group Leader in the organic chemicals laboratory of Dewey & Almy Division of W. R. Grace & Co., Cambridge, Mass.

Richard E. Benton, formerly of the Goodyear Tire & Rubber Co., is now Section Head in Coated Abrasives Research for Armour Alliance Industries, Alliance, Ohio.

G. Lamont Bidwell is now Vice-President and General Manager of the Riegel Paper Corp., Milford, N. J.

Stanford G. Blankenship has retired from Perkins-Goodwin Co. but will continue to serve the company in an advisory capacity.

Charles W. Carroll, formerly student at The Institute of Paper Chemistry, is now in the Operations Research Department of the Scott Paper Co., Chester, Pa.

Roy C. Charron, formerly of the U. S. Envelopes Co., is now an Adhesives Specialist for Stein, Hall & Co., New York, N. Y.

Jack E. Chinn is now Technical Supervisor of Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.

Orrin B. Clifford, formerly a Student at the University of Maine, is now an Engineering Trainee with Riegel Paper Corp., Milford, N. J.

Arthur A. Coffin of the Titanium Pigment Corp. has retired and will live in Glen Ellyn, Ill.

Ernest C. Cowell, formerly of West Virginia Pulp & Paper Co., is now with Allied Paper Corp., Chicago, Ill.

Cornelius A. Cunningham, formerly a Student at Lowell Technological Institute, is now a Chemist with National Starch & Chemical Corp., Plainfield, N. J.

William Deskins, formerly of the Fox Paper Co., is now a Consulting Engineer for the pulp and paper industry in Tampa, Fla.

Arden C. Dubert, formerly of West Virginia Pulp & Paper Co., is now

Assistant to the General Superintendent of the Oxford Paper Co., W. Carrollton, Ohio.

James S. Evans is now Vice-President and General Manager of the Wrenn Paper Co., Middletown, Ohio.

David W. Flotow, formerly of Hercules Powder Co., is now a Research Engineer for Consolidated Paper Co., Monroe, Mich.

Benjamin M. Foose, Jr., is now Manager of Industrial Sales for Riegel Paper Corp., Milford, N. J.

M. A. Hadi, formerly of Sirpur Paper Mills, is now Chief Chemist for Adamjee Paper Board Mills, Nowsberg, W. Pakistan.

Ralph S. Hartley, Physicist of Fiberboard Paper Products, Inc., has been transferred from San Francisco, Calif., to South Gate, Calif.

Sherrill J. Hazard, Jr., formerly student at the New York State College of Forestry, is now a project engineer for Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.

Phillip P. Holland, formerly a student at North Carolina State College, is now a Junior Technologist for Champion Paper & Fibre Co., Canton, N. C.

Stanley B. Huss, formerly of E. W. Twitchell, Inc., is now Assistant to the President of American Twine & Fabric Corp., Salmon Falls, N. H.

Donald T. Jackson is now Vice-President of the Hammermill Paper Co., Erie, Pa.

Robert J. Jacobs, formerly of the Black-Clawson Co., is now General Manager of the John Waldron Corp., New Brunswick, N. J.

Thomas M. James, Jr., formerly of the Flox Co., Inc., is now a Service Engineer for Nalco Chemical Co., Portland, Ore.

John B. Kahoun, formerly of the Nekoosa Edwards Paper Co., is now a Designer for the Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.

Frederick C. Keeney, formerly of Weyerhaeuser Timber Co., is now Assistant to the Vice-President in Charge of Production of Knowlton Brothers, Watertown, N. Y.

Phillip L. Knapp, formerly of Bulkley, Dunton Co., Inc., is now Vice-President of the Butler Co., Chicago, Ill.

Engelbert Krichels is now President of the Hurlbut Paper Co., So. Lee, Mass.

Charles A. Lee is now General Manager of the Formex Co., Division of Huyck Felt Co., Knoxville, Tenn.

Edward G. Locke is now Director of the Forest Products Laboratory, Madison, Wis.

E. David McKay is now Assistant Research Director for the Southern Chemical Cotton Co., Chattanooga, Tenn.

Jerome C. Marquard of the Marathon Division of American Can Co. has been transferred from Menasha, Wis., to Oswego, N. Y., as Assistant Paper Mill Superintendent.

Rex Morris of Crown Zellerbach Corp. has been transferred from Lebanon, Ore., to Camas, Wash., as Assistant Resident manager.

L. A. Moss is now Manager of Manufacturing Services for Riegel Paper Corp., Milford, N. J.

T. Richard Probst is now President of the Deerfield Glassine Co., Monroe Bridge, Mass.

Hugh P. Quinn, formerly of Ross Midwest Fulton Corp., is now on the Technical Service Staff of the Lindsay Wire Weaving Co., Cleveland, Ohio.

George H. Rand is now a member of the Executive Staff of the International Paper Co., New York, N. Y.

Nicolas M. Reitzel, formerly of the Rice-Barton Corp., is now a group leader for the West Virginia Pulp & Paper Co., Covington, Va.

Walter J. Rew, formerly of the West Virginia Pulp & Paper Co., is now a project engineer for the Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.

Bruce St. Ledger, formerly a student at the University of

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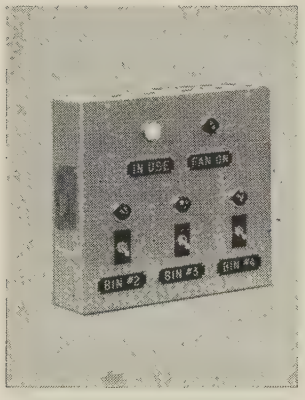

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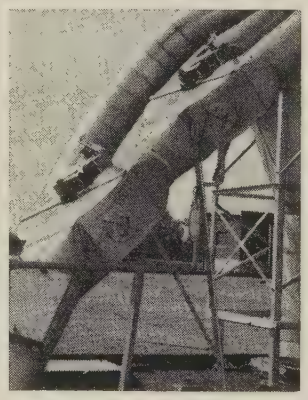

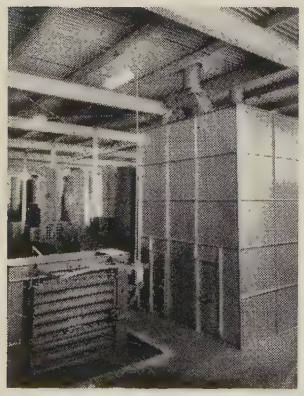

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Maine, is now a production trainee for the Container Corp. of America, Manayunk, Pa.

W. Allan Schenk is now Manager of Paper Production for the Riegel Paper Corp., Milford, N. J.

William L. Schnorback is now Plant Manager of the Continental Can Co., Augusta, Ga.

F. Keister Smith, formerly of the West Virginia Pulp & Paper Co., is now a Crating Specialist for the Champion Paper & Fibre Co., Hamilton, Ohio.

Ronald R. Smith, formerly of the Gardner Board & Carton Co., is now Staff Engineer for the Beloit Iron Works, Beloit, Wis.

Ashworth Stull is now Assistant Vice-President for Technical Service for the Borden Chemical Co., North Andover, Mass.

Peter P. Taranoff is now Paper Machine Superintendent for the Crown Zellerbach Corp., St. Helens, Wash.

Rex A. Thorne of the Union Bag-Camp Paper Corp. has been transferred from Chicago, Ill., to Jamestown, N. C., as Plant Manager for the Highland Containers.

John E. Ward of Nopco Chemical Co. has been transferred from Newark, N. J., to Nopco Chimie, S/A Fribourg, Switzerland.

Alvin R. Wilson is now Technical Director of Le Page's, Gloucester, Mass.

Donald A. Yateman, formerly of Dixie Cup Co., is now research chemist for P. H. Glatfelter Co., Spring Grove, Pa.

Charles E. Young is now Converting and Shipping Superintendent for Crown Zellerbach Corp., St. Helens, Ore.

Frank Zeitlin, formerly of Alton Box Board Co., is now Assistant Vice-President of Mallinckrodt Nuclear Corp., St. Louis, Mo.

Industry Notes

Production

Production of paper and paperboard in the first half of 1959 is estimated at 17 million tons. This established a new high for the first half of the year, and was about 14% above the total produced in the first half of 1958. Production of paper in the first half of 1959, at 7.4 million tons, was 11% above the year-ago total; paperboard production, at 7.9 million tons, was 15% above the year-ago total. The relatively high rates of residential construction activity in the first half of 1959 brought production of construction paper and board to a rate 17% above the year-ago level (APPA figures).

Paperboard production for the first six months of 1959 was 16.6% greater than the comparable period last year, Albert W. Luhrs, executive manager, National Paperboard Association, has announced. Average weekly shipments of this essential material used in all types of packaging through June 27 was 306,700 tons compared with 263,100 tons during the same period of 1958. Second quarter production was 6.8% higher than the first quarter of 1959.

If the present rate continues 1959 will be a record year with 15.9 million tons of paperboard produced, Mr. Luhrs declared. The previous record year was 1956 when the paperboard mills turned out 14.4 million tons.

Pulpwood

Total receipts of pulpwood for the first five months of 1959 were 14,908,000 cords, an increase of 1,171,000 cords over receipts for the comparable period of 1958. Domestic receipts were 1,240,000 cords above the year earlier total, while imports declined 78,000 cords. All of the regions showed increases, ranging from 3% in the West, to 12% in the South Atlantic.

Pulpwood consumption in the first five months of 1959 was 15,959,000 cords, an increase of 1,543,000 cords over the same period of 1958. The Northeastern region showed a 1% decline in consumption while all other regions showed increases, ranging from 5% in the West to 16% in the South Atlantic region.

Inventories of pulpwood at the end of May were 4,852,000 cords, a decrease of 353,000 cords below April, 1959, and a 18% decline from May last year. All of the regions showed decreases, ranging from 5% in the South Central to 32% in the West.

Wood Pulp

Production of wood pulp for the first five months of 1959 was 9,991,000 tons, an increase of 1,191,000 tons above the comparable 1958 total.

Wood pulp consumption in the manufacture of paper and board for the first five months of 1959 was 10,335,000 tons, an increase of 1,289,000 tons above consumption for the first five months of 1958.

Imports of wood pulp for January—May, 1959, were 996,908 tons, an increase of 26% above imports for the first five months of last year. Imports from Canada for the first five months of 1959 were 811,727 tons, an increase of 17% over the comparable period of 1958. Overseas imports for the first five months of 1959 were 182,457 tons as compared to 98,946 tons in the first five months of 1958.

Exports of wood pulp for the first five months of this year were 233,816 tons, as against 224,134 tons exported during the same period last year.

Inventories of wood pulp at paper and board mills at the end of May were 556,000 tons, an increase of 11,000 tons above inventories at the end of April, but 4000 tons below inventories at the end of May, 1958.

Wastepaper

May wastepaper receipts were 802,000 tons; for the first five months of this year they totaled 3,876,000 tons, an increase of 433,000 tons above receipts for the first five months of last year.

Wastepaper consumption for May was 784,000 tons; the January—May total was 3,824,000 tons, an increase of 332,000 tons above consumption for the first five months in 1958.

Inventories at the end of May were 489,000 tons, an increase of 17,000 tons above April, but 1000 tons below inventories at the end of May, 1958.

Consumption of total fibrous materials for May was 874,000 tons; the January—May total was 4,268,000 tons, an increase of 1,048,000 tons above consumption for the first five months of 1958.

Inventories of total fibrous materials at the end of May were 898,000 tons, 7000 tons below April, and 14,000 tons below inventories at the end of May, 1958.

Fiber Boxes

Shipments of corrugated and solid fiber boxes which move the Nation's goods from farm and factory to consumer were 15.9% higher during the first 6 months of 1959 than the same period last year, Albert W. Luhrs, executive manager, Fiber Box Association has announced.

Fiber box plants shipped a record average of 2.05 billion sq. ft. each week, during the first half of 1959 as compared to 1.77 billion sq. ft. in the same period of 1958. Should this rate of production continue throughout the balance of the year, Mr. Luhrs said, total shipments of corrugated and solid fiber boxes in 1959 will reach 108 billion sq. ft. This would be an 11% increase over the previous record set in 1956 when 97.6 billion sq. ft. were shipped.

Finland

Kymmene Ab has ordered from Valmet Oy for Voikka Paper Mill a new high-speed newsprint machine with a working width of 518 cm. trim, a constructional speed of 800 m. per min., and an annual production of 70,000 tons. Differential drive for the machine, winder, auxiliary machines, air conditioning and installation are included in Valmet's delivery. Time of delivery is about two years.

Waste paper furnish grades of corrugating medium take on added strength when General Mills versatile GUARTEC gum is added to the furnish. This increased stiffness is apparent both with and across machine direction! GUARTEC disperses quickly in both continuous and batch cooking systems. It is used as straight gum sol and in cooked gum-starch blends.

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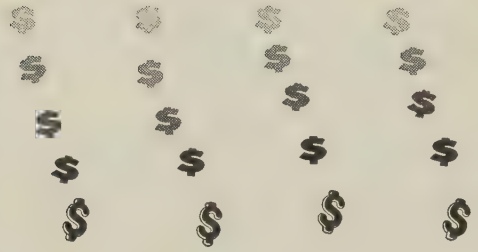

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Stainless Impellers

Subjected to punishing corrosion and erosion, liquor-circulating pumps with cast stainless steel impellers have operated efficiently for 9 years in chemical pulp processing at The Mead Corp.'s Kingsport Div., Kingsport, Tenn. Mead engineers standardized on cast impellers made from alloy type CF-8M (Alloy Casting Institute designation) to resist the corrosive solutions handled in alkali-recovery pumping operations. Pedestal-mounted, end-suction, overhung impeller type pumps are used throughout and, as yet, the impellers show no signs of corrosive attack or wear.

In addition to the corrosive nature of the liquors handled, high rates of flow through the impeller tend to cause erosion by high velocity impingement, and accelerate the corrosive action of the fluids. To resist these severe conditions, the CF-8M cast stainless alloy (19% Cr, 10% Ni, 0.08% max C, 2.0 to 3.0% Mo) was selected for the impellers of the battery of pumps. This grade is noted for its exceptional corrosion resistance and strength.

Surface Flammability

Comprehensive new information on how rapidly flame spreads across the surface of lumber, plywood, hardboard, particle board, and fiberboards is brought out for the first time in a new report just issued by the United States Forest Products Laboratory, Madison, Wis.

Under the title, "Surface Flammability of Various Wood-Base Building Materials," the report, No. 2140, presents data on 29 species of wood and 50 commercially produced sheet materials. The authors, Chemist H. D. Bruce and Engineer L. E. Downs, characterize the results as "providing a realistic relative measure of the surface flammability of the various materials tested."

The tests were conducted in the laboratory's newest equipment, a small tunnel furnace specially designed for this work. The results, the authors point out, correlate well with earlier wall-corner tests simulating room fires, which had been conducted on 11 different materials. The FPL tunnel furnace has recently been accepted by the American Society for Testing Materials as a tentative standard test.

Surface flammability, according to the authors, is regarded by fire protection engineers as an important property of a building material.

All materials tested were rated on the basis of a rating of 100 for oak lumber and 0 for asbestos board. All were first conditioned to dry air kept at a uniform temperature of 80°F. and 30% R.H.

The lumber tested consisted of boards nominally 1 in. thick. Softwood and hardwood plywoods were tested in $^1/_4$- and $^3/_8$-in. thicknesses, and the other materials in various commercial thicknesses.

Besides flame spread, the materials were also rated for density of smoke given off and amount of heat contributed.

Single copies of the report are available free upon request to the director. Also available is a newly revised list of publications on fire protection listing a number of the laboratory's reports in this field as well as other literature.

The laboratory has also issued a new report on the veneer cutting and drying properties of redwood, Report No. 1766-8. Newly revised are its Report No. 446, "List of Publications on the Seasoning of Wood," and Report No. 564, "Partial List of Reference Works on Pulp and Paper." All of the aforegoing publications are available free of charge from the Director, Forest Products Laboratory, Madison 5, Wis.

Colored Stock

Creative use of colored paper stock as a basic printing tool can result in more effective and eye-stopping printed pieces but lack of knowledge about, or confidence in, the techniques and effects of using colored inks on colored stock has hampered its widespread adoption, says the Du Pont Co.

Basing its viewpoint on a nation-wide survey of artists,

art directors, and commercial printers, the company's Dyes and Chemicals Division, major supplier of quality dyes to the paper industry, finds that while 98% of the printers polled have used colored stock to some extent, they may be overlooking increased business and income by failing to promote it more extensively. Du Pont's solution: an educational campaign enabling paper suppliers to show printers, by actual printed samples, how colored stock can be handled and the "bonus" effects it can contribute to modern printing.

Drawing upon the combined talents of artists, advertising consultants, printing experts, and color chemists, Du Pont has assembled for its paper supplier customers a variety of typical printed pieces utilizing colored stock. Also included is a guidebook for color printing practice, showing the many variations in effects obtained when colored paper is used as a base for printed text, tints, overlays, halftones, and duotones.

Only 7% of all the printers contacted in the company's survey said that colored stocks accounted for more than half of their paper requirements. Twenty-nine per cent estimated their usage of colored paper at 26 to 50% of total requirements, and 12% stated colored stock accounted for 21 to 25% of their total. At the other end of the scale, 21% of the printers estimated their colored stock requirements at 5% or less of total paper requirements.

Use of colored stock is increasing in the opinion of 56% of the printers interviewed. Factors responsible for the increase, they say, range from such things as "competition today forces eye-catching color" to an increasing realization on the part of customers of the sales appeal of messages printed on colored stock. About 17% of the printers credited widespread adoption of multicolored business forms with the boost in color stock usage.

Among other findings in the survey, Du Pont said 75% of the printers indicated a feeling that black and white photographs do not look as good on colored stock as on white stock. Seventy per cent also noted that "customers are often disappointed at the way inks show up on colored stock," 61% stated "it is more difficult to match an engraver's proof on colored stock than on white," and 60% said that better results, from the printer's point of view, are attained by press-imprinted color rather than "starting with a colored stock." When actual samples of printing on colored stock were shown and discussed with the printers, however, many were pleasantly surprised with effectiveness of the results.

Essentially, the same basic reasons for minor use of colored stock were mentioned by artists and art directors interviewed in the Du Pont survey.

Many of the feelings about colored stock expressed by interviewees, Du Pont's Dyes and Chemicals Division said, are simply the result of habits, based on part upon the fact that so much of their work has been on white paper rather than colored stock. Advantages of colored paper as a basic starting tool simply haven't been impressed on them, and, consequently, they just don't think of using colored paper.

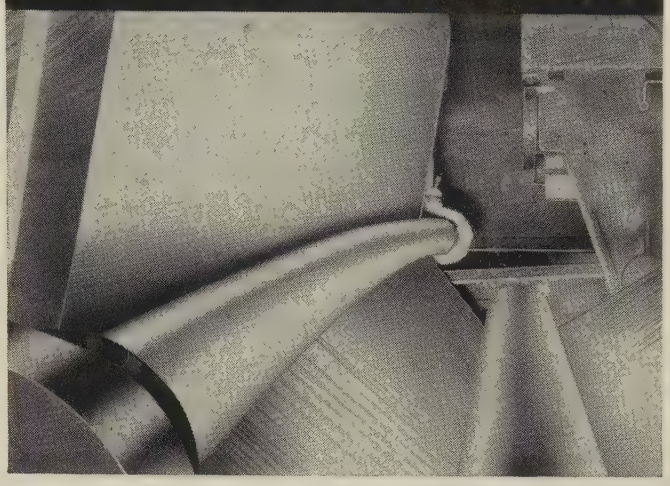
Southern Pulp

More than 20 million cords of pulpwood were harvested in the South during 1958, according to a report of the Forest Service of the United States Department of Agriculture. This production was 2% more than that of 1957, and represented 58% of all wood used by United States pulp mills during the year.

The report gives detailed information on the South's 1958 pulpwood harvest, and reviews trends since 1946.

The capacity of Southern pulp mills has increased steadily since World War II, and now stands at a record high of over 44,000 tons per day. At the end of 1958, the South had 75 pulp mills, representing three fifths of the Nation's pulping capacity. Five new mills are in prospect. The number of southern mills producing newsprint rose from 1 in 1946 to 5 in 1958.

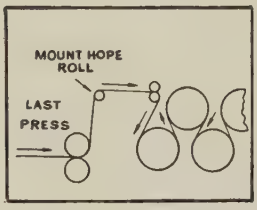
PICTURE OF A MILL STRETCHING ITS PROFITS . . .

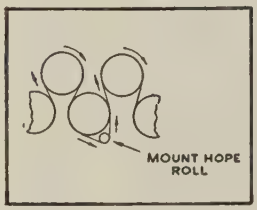


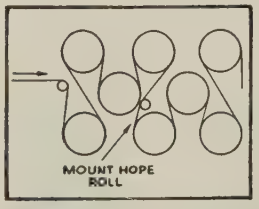
by STRETCHING-the-SHEET with

MOUNT HOPE ROLLS

3 TYPICAL INSTALLATIONS







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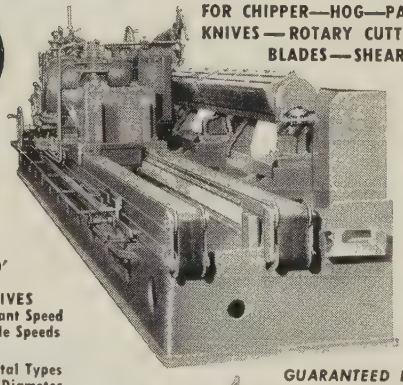

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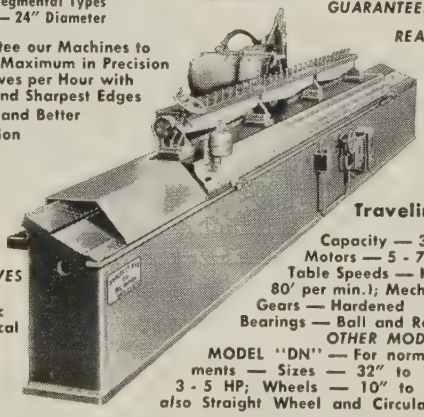

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Georgia, the South's leading producer of pulpwood, harvested 4 million cords in 1958. Alabama ranked second, with 2½ million cords. North Carolina was third, with almost 2 million.

Baldwin County, Ala., produced more roundwood than any other county—135,000 cords. Others cutting more than 100,000 cords were Union Parish, La.; Camden, Ware, and Wayne Counties in Georgia; Union County, Ark.; and Fairfield County, S. C.

Almost 10% of the wood used by the mills is now salvaged from the residues of other wood-using plants—chiefly from the slabs and edgings at pine sawmills. Until 1952, such material was largely wasted. Its increasing use, the report states, is one of the most notable developments in the industry.

Southern pine continues to be the most important wood in the nation's pulp industry, furnishing about half of all wood, domestic and foreign, consumed by United States pulp mills in 1958. The cut of southern hardwoods is steadily rising. The 1958 hardwood harvest, 3 million cords, set a new record for the eighth consecutive year. Some hardwood is cut in every southern State, but Mississippi is the leading producer.

The report, titled "Southern Pulpwood Production, 1958," was prepared by the United States Forest Service in co-operation with the Southern Pulpwood Conservation Association, Atlanta, Ga. Copies are available from the Southeastern Forest Experiment Station, Asheville, N. C., or from the Southern Forest Experiment Station, New Orleans, La.

The following table shows pulpmill capacity and amount of wood produced by state.

Pulpwood Production by States, 1958

State	Number of mills	Mill capacity in tons of pulp per day	Pulpwood produced, thousand cords		Three leading counties
			Roundwood	Residues	
Ala.	9	4103	2370	254	Baldwin, Monroe, Washington
Ark.	4	2115	1041	273	Union, Ashley, Clark
Fla.	10	8100	1752	83	Marion, Nassau, Baker
Ga.	10	6750	3861	232	Ware, Camden, Wayne
La.	10	5605	1438	146	Union, Winn, Washington
Miss.	6	2962	1786	99	Clarke, Jasper, Jones
N. C.	5	3810	1706	190	Buncombe, Onslow, Columbus
Okla.	1	90	47	16	McCurtain, LeFlore, Mayes
S. C.	3	3530	1593	155	Fairfield, Georgetown, Newberry
Tenn.	4	1832	349	6	Monroe, Hawkins, McMinn
Texas	5	2305	1127	259	Montgomery, Nacogdoches, Shelby
Va.	8	3300	1378	72	Amherst, Buckingham, Alleghany
Total	75	44,502	18,448	1785	

New C-Flute

Owens-Illinois Glass Co. is sharing with the corrugated box industry the fruits of a five-year research and development program that resulted in the perfection of a new, stronger, type of C-flute corrugated board.

The new board, called O-I C-flute, achieves up to 17% greater resistance to flat crush than ordinary C-flute through a change in the design of the flute contour.

Now, benefits of O-I C-flute are being made available to other companies in the box business.

The S. M. Langston Co., who designed the original roll cutters required to make the special corrugating rolls, has been directed to use the O-I cutters in making rolls for any company that wishes to use them. A substantial number of

orders for the rolls have been received, according to Langston officials.

A basic research and development study of flute contours was launched by Owens-Illinois in 1955 under the direction of Richard O. Spalding, coordinator of corrugated operations for O-I, and F. W. Wallis, now vice-president-manufacturing, O-I Paper Products Division.

Past operating experience indicated that the program should start with a complete study of the problem on the Concora medium tester.

Months of comparing the design and performance of a great many modifications of C-flute—experimenting with different numbers of flutes per foot, different thicknesses of board, and different contours—indicated that C-flute could be strengthened appreciably by redesigning the flute contours.

The result of this study, after thousands of tests, was O-I C-flute—with a "contour engineered" design that yielded an actual CMT reading of 56.91 p.s.i. against 48.58 p.s.i. for conventional C-flute, a 17.14% improvement.

Added strength, however, was but one of the outstanding features of O-I C-flute; a number of other major improvements were encountered as direct by-products of this development.

General runability is better because there are fewer high and low corrugations with rigid semichemical corrugating mediums; with conventional C-flute this had been one of the major corrugator operating problems.

The printing surface is substantially improved because the board is more rigid. For the same reason, the board permits more positive folding during automatic assemby operations.

Dynamic cushion, Conbur impact, compression Conbur crush, bottom drop and vibration tests indicated that O-I C-flute performed as well as, or better than, conventional C-flute.

Turbine Aeration

Eight tons of dissolved oxygen are being added to Wisconsin's Fox River every day between Kimberly and Little Rapids through turbines that have been specially modified for stream improvement by four pulp and paper mills, the Pulp & Paper Information Service has announced. This exceeds 20% of the 38 tons of oxygen that state pollution authorities calculated would restore the Fox River to a satisfactory oxygen balance after their most recent survey of that stream.

Turbine aeration is a newly developed technique that has been experimentally tested and approved by Wisconsin pulp and paper industry researchers during the past three years to replace oxygen in streams whose oxygen content has fallen below desired levels. It has been put to work by several Wisconsin mills to supplement their active programs for collecting and utilizing oxygen-consuming materials which formerly were discarded into rivers. Authorities expect that it will be adopted at more and more power plants on streams where higher proportions of dissolved oxygen are needed.

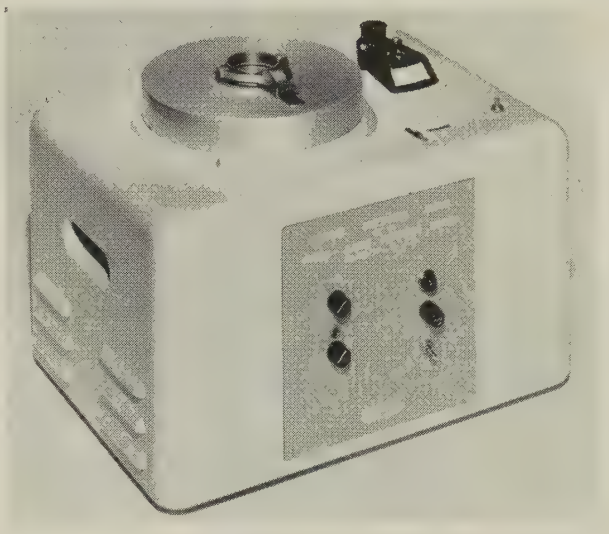
Mills currently utilizing turbine aeration on the lower Fox are: Charmin Paper Products Co., Little Rapids; Combined Locks Paper Co., Combined Locks; Kimberly-Clark Corp., Kimberly; and Thilmany Pulp and Paper Co., Kaukauna.

Nonwovens

Production of nonwoven fabrics may reach a record 125 million pounds this year, *Chemical and Engineering News* estimates, on the basis of half-year production figures now coming in. The previous high was 110 million pounds in 1957. Ten years ago annual production of nonwovens was less than a million pounds.

New fibers, synthetic binding materials, and improved webbing patterns, created by chemists and engineers, have

FOTOSIZE LIQUID PENETRATION TESTER



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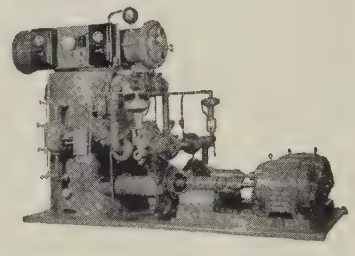

The Stickle Vacuum Pump is made exclusively for paper mills. It's easy to service. All floats housed in exterior float boxes. Interchangeable, removable vacuum chamber is above tank. Inspection of ejector tubes may be made without moving motor, pump or piping. Uses standard horizontal motor and pump. For vacuums to 28". Built-in heat exchanger eliminates costly cooling equipment. Recirculation of hurling water condenses flash vapors and releases air and gases to atmosphere. No close clearances to cause wear. Performance guaranteed. Single and duplex models. Write for free literature.

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established this new beachhead for the industry, which now hopes to invade some bordering market areas held by textiles and paper products. Many experts agree that total production could easily triple within three years if nonwovens could gain a place in wearing apparel, says the American Chemical Society weekly. The 10-million-pound slump in 1958 is not considered a trend because fall-off in production merely followed that of fabrics in general.

At the outset, a nonwoven fabric simply meant a cheap product made largely from low grade and waste cotton fibers laid down in a web or continuous sheet and held together by low cost binders, such as starch, glue, and gums. Rug underlays are an example. Now the picture is changing in several ways.

Present advances into the clothing market have put an estimated 10 million pounds of nonwovens—mostly as interlining—into ties, suits, dresses, foundation garments, quilted garments, handbags, and shoes, it is stated. Disposable and semidurable items such as wrap-around garments for laboratory and industrial use, party costumes, and aprons are being made of nonwoven materials. Durable nonwoven skirts and dresses are on the market.

Researchers are working hard on technological improvements to give nonwoven fabrics better drape and feel and increased durability to laundering and drycleaning in the hope of gaining public acceptance for durable nonwoven outerware, the magazine reports. But the general use of disposable outer garments is not considered too promising in view of high tailoring and distribution costs.

Nonwoven home products such as draperies, curtains, ticking, covers, towels, polishing cloths, and lampshades are catching on. Dental and medical applications are on the upswing. And shoe manufacturers are increasing their orders for nonwoven innersoles and interliners. Nonwovens are being used in cowboy hats, doll dresses, tea bags, and diapers.

Regardless of whether nonwoven fabrics move in on markets for apparel or paper products, consumption will increase this year, says *Chemical and Engineering News*. The biggest immediate potential is in industrial applications, where observers predict a demand for 70 million pounds in 1959. Uses include automobile quilting and padding applications, backing for plastic sheeting, laminates, filters, wiping cloths, insulation, bags, and packaging materials.

About 60% of the fibers now used in nonwoven materials are synthetics, some of them self-binding types, the magazine notes. About 30 million pounds of viscose rayon and about 12 million pounds of acetate and waste nylon fibers were used last year.

In addition to the old binders, such as starch, gums, and casein, synthetic resin binders and rubber and plastic latexes are now used. Cotton Christmas tree "snow" contains less than 10% binder, while shoe innersoles are made with three times as much binder as fiber. About 28 million pounds of binders and 2 million pounds of thickeners, plasticizers, pigments, wetting agents, fillers, and chemical agents for resistance to fire and water are used in a year.

Many of the problems hampering production growth of nonwovens fall into the laps of the chemist and the engineer, the magazine comments, but these problems are being solved by research.

"Before nonwovens could succeed in certain markets, particularly in apparel areas, they needed new and improved properties like resiliency, crease resistance, drapability, hand (feel) and good wet and dry cleaning performance," explains *Chemical and Engineering News*.

This need ushered in new types of latex binders. Butadiene-styrene types led the way but were badly handicapped by poor stability to light and poor wet and dry cleaning properties Producers quickly moved on to butadiene-acrylonitrile latexes.

"But here they found crosses to bear, too. Nonwovens containing butadiene-styrene binders often discolor and give off odors when they age. Moreover, some binder migrates to the surface during bonding to leave the fabric weak in the center and prone to delaminate. While binder suppliers have done much to remedy odor and lightfastness problems, migration remains a big headache.

"Now, attention is swinging to acrylate binders. Acrylic esters, which really didn't get off the ground until last year, are making a strong bid for nonwoven markets. While these binders are not as elastic as butadiene-acrylonitrile types, they have good color and odor stability."

There are problems at the merchandising level also. The marketing is rough, says the magazine, adding:

"The textile industry is notorious for its fiercely competitive business practices, and its newest offspring, nonwovens, seems to have inherited most of the bad traits."

Celanese

One of Canada's richest undeveloped areas of natural resources was opened for commercial utilization in June with the inauguration of regular log towing service between the timberlands bordering the glacier-fed Nass River in northwestern British Columbia and Prince Rupert, B. C.

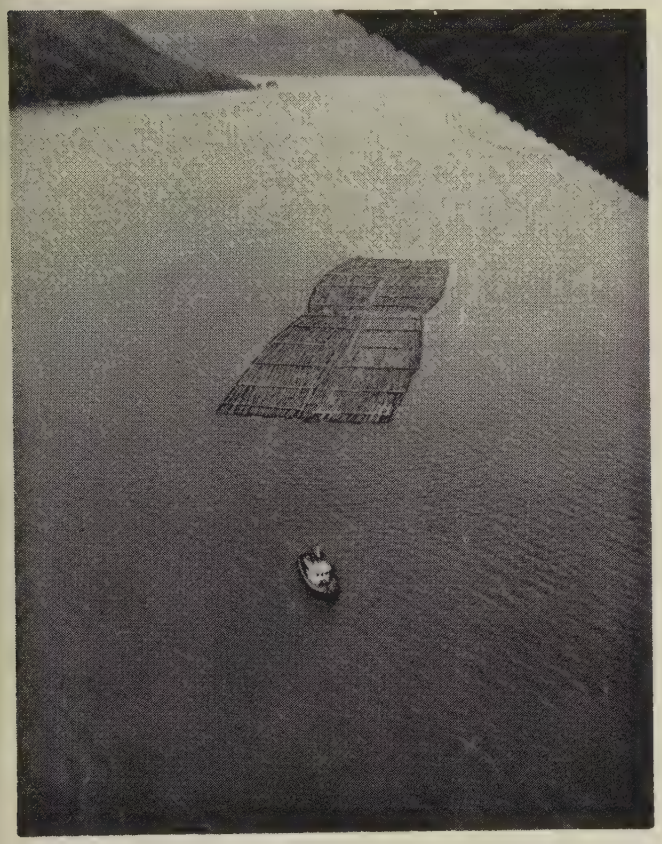
Sea-going tug boats haul approximately one million board feet of flat-rafted logs per trip on the two-day, 72-mile coastal voyage from the mouth of the Nass River to Prince Rupert where the logs are delivered to the sulphite pulp mill owned by Columbia Cellulose Co., Ltd., an affiliation of Celanese Corp. of America. At the mill, the logs are converted into dissolving wood pulp—the basic raw material used in the production of acetate and rayon fibers, acetate plastics, and specialty grade papers.

Towing service on the previously isolated Nass River is now on a daily basis. Shallow-draught tugs make two trips daily from the booming grounds, 35 miles up the Nass, to the river's

mouth where the single flat-raft or boom is made up into a larger, four-boom tow for pickup by the sea-going tug. On the river tows, one tug pulls the boom and the other steers it from behind. Only one tug is used for the deep-sea tow.

Vast stands of virgin timber and abundant but still untapped mineral resources are included in the Nass River's 7,000,000-acre watershed. However, due to its former inaccessibility, this nature-rich area has contributed practically nothing to the economic life of British Columbia. Until late last year, there were no roads within a 70-mile radius. And water transportation was limited to canoes and a few small power boats owned by the scattered bands of Tsimshian Indians who live along the banks of the Nass.

The first vehicular link between the Nass Valley and inhabited areas of British Columbia was opened last October with



Deep-sea tug boat, en route to pulp mill at Prince Rupert, pulls a four-boom tow (four river booms lashed together) containing approximately 1 million board feet of logs. The tug makes the 72-mile coastal voyage from the Nass to Prince Rupert in 2 days. At the Columbia Cellulose mill, the logs are converted into dissolving wood pulp

the completion of a 70-mile stretch of all-weather road built by Columbia Cellulose to gain access to its timber holdings in the Nass River area. The road runs from the company's main logging camp at Terrace to a point on the Nass River some five miles west of the Indian village of Aiyansh. With the opening of the road, the heavy mobile equipment required for large volume logging operations was moved north toward the Nass. This, in turn, made possible the establishment of log towing service on a regular commercial basis from the Nass River to Prince Rupert.

Simpson Lee

Merger of the pioneer Simpson Paper Co. of Everett, Wash., with the Lee Paper Co. of Vicksburg, Mich., to form a new Washington corporation to be called the Simpson Lee Paper

Chemical Pulping Processes

The high degree of chemical, mechanical and physical control over materials and operations in the Chemipulp and Chemipulp-KC systems results in maximum plant output, high pulp quality and low operating costs.

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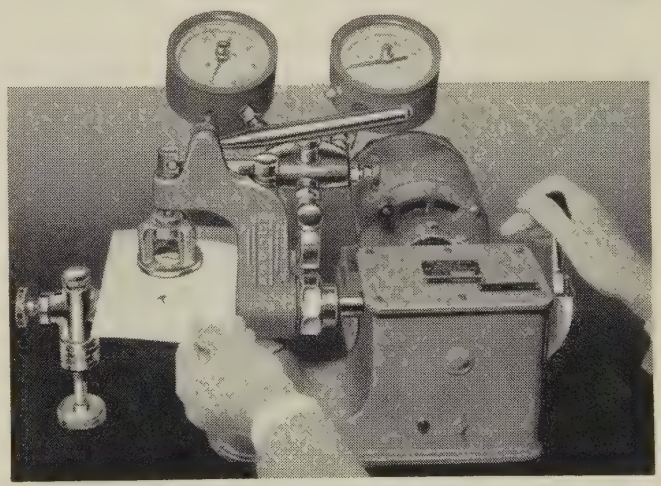

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Co. was announced jointly August 6 by Thos. F. Gleed, president of Simpson Timber Co. in Seattle, and Maxwell D. Bardeen, president of Lee Paper Co. in Vicksburg.

Simpson Paper Co., founded in 1891, operates fully integrated pulp, paper, and converting plants in Everett as well as a branch converting plant in Los Angeles. It is a wholly owned subsidiary of Simpson Timber Co.

W. G. Reed, chairman of Simpson Timber Co., will be chairman of the new Simpson Lee Paper Co. He has been a major stockholder and director of Lee Paper Co. for a number of years.

Serving as directors with Reed will be Mr. Gleed, C. H. Bacon, Jr., and C. H. Kreienbaum, executive vice-president and vice-chairman, respectively, of Simpson Timber Co., all of Seattle; Maxwell D. Bardeen, Norman Bardeen, vice-president in charge of sales of Lee Paper Co., Merril W. Taylor, banker, and John C. Howard, lawyer, all of Kalamazoo, Mich.

Maxwell D. Bardeen will be president of Simpson Lee Paper Co. Vice-presidents will include Donald F. McCall, now vice-president and general manager of Simpson Paper Co., Norman Bardeen and Albert A. Christian, the latter now general manager of Lee Paper Co. Mr. Christian will become secretary of the new company. Kenneth A. Warren, treasurer of Lee Paper Co., will be treasurer of Simpson Lee Paper Co.

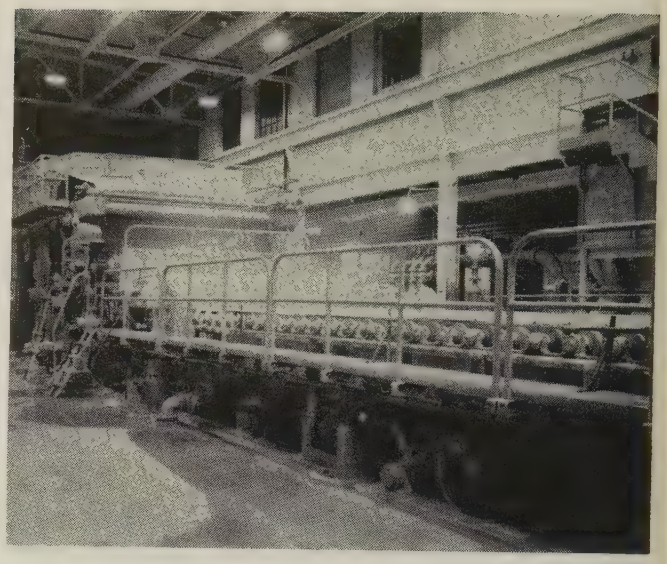
In explaining merger plans to Simpson Lee Paper Co. supervisors and employees, Mr. McCall stated that this progressive combination provides strengthened management, assets, and resources, and expanded product lines for Simpson and Lee customers. Present product lines of the two companies do not conflict with each other. Lee produces cotton rag content and technical and specialty papers which complement most advantageously the fine papers produced by Simpson including writing, book, label, and offset grades, both coated and uncoated.

Additional details concerning the merger and future plans for the Simpson Lee Paper Co. will be announced as arrangements progress and are completed.

Lee Paper Co., which employs 350, was established in Vicksburg, 15 miles south of Kalamazoo, in 1904. The father of Maxwell Bardeen was among the key employees of the original mill. A graduate of Yale University, Mr. Bardeen has worked in all parts of the company. He served as mill superintendent until his father's death in 1936 when he became general manager and, later, president.

A singular salute from industrial and business neighbors and suppliers was accorded Simpson Paper Co. on May 29 when the *Everett* (Wash.) *Daily Herald* published a special edition on the theme "Simpson Grows in Everett." Consisting of 48 pages in four sections and utilizing their full-color reproduction facilities, the newspaper paid special tribute to the community's oldest continuously operated industry with particular emphasis on recent modernization and expansion programs involving investments in excess of $4 million. The entire edition was printed on a modified mimeo-bond paper produced by Simpson especially for the occasion.

Prominently featured were pictures and articles on the completely new, modern high-speed paper machine installed by the Everett plant late last year. Designated by Simpson employees with a name of particular Northwest flavor, The Royal Chinook, the new machine replaced the company's largest no. 3 paper machine which had been installed in 1926. That machine was completely dismantled and removed and the modern equipment erected in the three-story brick building in a record time of only 47 days. The Howard S. Wright Co. of Seattle was general contractor in charge of the physical removal of the old machine and erection of the new equipment furnished by the Beloit Iron Works of Beloit, Wis., as prime supplier.



Simpson's newest paper machine, The Royal Chinook, is capable of mechanical speeds up to 1500 f.p.m. This is double that of the old no. 3 machine which was completely dismantled and removed to make room for the new equipment. The 155-in. wide wire on the fourdrinier section shown here has an overall length of 105 ft., 8 in. as compared with the former wire length of 70 ft.

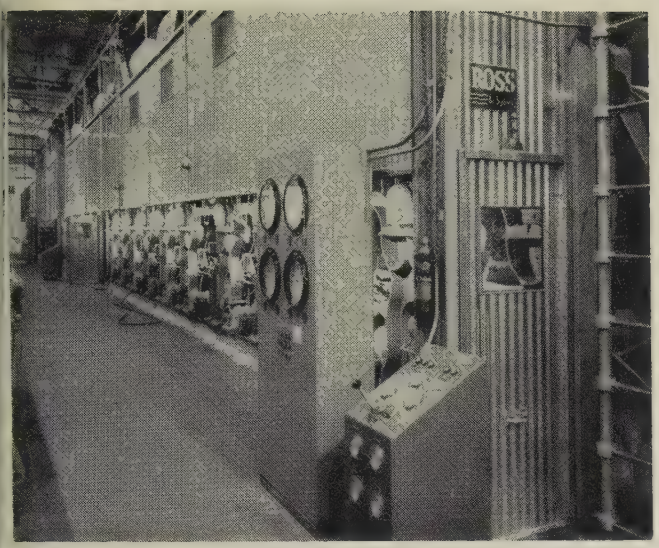
Simpson's production and engineering staffs together planned the project and accomplished the general design work. Participants, in addition to M. W. Olson, resident engineer, and his senior project engineer, John Gould, included W. A. McKenzie, formerly resident engineer and now general manager of Simpson Engineered Wood Products Co., H. R. Russell, paper mill superintendent, and F. R. Hamilton, director of research and product development. Detailed engineering was accomplished by H. A. Simons, Ltd., of Vancouver, B. C., engaged as consulting engineers for the project.

D. F. McCall is vice-president and general manager of the paper company and P. F. Peterson, plant manager. Established in 1891, the company was formerly known as the Everett Pulp and Paper Co. prior to its purchase in 1951 by the Simpson Timber Co. of Seattle. W. G. Reed is chairman of the board of the parent company and T. F. Gleed, president.

The Royal Chinook is capable of a mechanical speed of 1500 f.p.m., twice that of the old machine. The 155-in. wide fourdrinier wire which had previously been 70 ft. in length gave way in the new model to an over-all length of 105 ft., 8 in. Additional drying capacity required by the faster operating speeds demanded an increase in over-all machine length to 270 ft. to accommodate 48 60-in. drier cylinders in lieu of the former 31. First and second drier sections are totally enclosed by Ross-Hooper insulated aluminum hoods. Designed specifically for production of highest quality book, writing, offset, and label papers, it is the only machine of its type on the Pacific Coast with fully enclosed driers. The sides, which extend to the floor and permit faster drying and more accurate humidity control, are both automatically and manually operated.

Immediately following the normal suction press, an inverted suction press has been added to reduce the tendency toward two-sidedness by withdrawing excess water from both sides of the sheet rather than just from the underside as formerly.

A new breaker stack has been installed between the first two drier sections and a new size press is located between the second and third drier sections. The latter is immediately preceded by one of the former machine's calender stacks,



With the sides of the insulated aluminum hood which fully encloses the first two drier sections of Simpson's new Royal Chinook in the raised position, a portion of the 48 60-in. drying cylinders which replaced the former 31 on the old machine can be seen. The overall length of the new machine was extended to 270 ft. to accommodate the greater drying capacity required by the faster operating speeds and for additional equipment such as inverted suction press and size-coating press

completely rebuilt to conform to the greater speeds and size of the new machine. This stack provides a smooth surface finish prior to the application of sizing or coating.

The entire machine is powered by a single 1000-hp. General Electric turbine transmitting energy through a Beloit differential lineshaft drive to 11 takeoff points controlling the various sections of the machine.

Automatic control of the drives as well as attendant operations of the machine itself is accomplished through a pneumatic control system and photoelectric cells. Additional automatic monitoring devices throughout the machine utilize pressure, level, temperature, and electrical sensing and transmitting equipment. Electrical installations were accomplished by Rogers Electric Co. of Everett in conjunction with plant electricians under the direction of Harold Thoreen, chief electrician, and Bill Andrews, Simpson's electrical engineer. Subcontractor for piping was the Haskell Corp., also of Everett.

A battery of 15 Bauer centrifugal cleaners precede four Black-Clawson Selectifier screens and a Beloit pressure headbox. The latter replaced the open headbox of the former machine and permits uniform control of stock flow by increasing or decreasing pressures in the headbox.

The limitation in the length of the building erected originally to house the old machine required that the Cameron slitter and rewinder be placed to the side of the final windup reel. This winder is equipped with a new Beloit Tru-Tension device and brake. Overhead traveling cranes swing the machine rolls in a 90° angle to the slitter and rewinder which is installed in an opening in the machine room wall resulting in rewound rolls being delivered on the finishing room side where they are easily transportable to supercalender stacks, rotary cutters, embosser, or to Simpson's high-speed air-blade off-machine coater.

One of the first units installed for the new machine and actually erected before the old equipment was removed was a Liebeck broke pulper produced by the E. D. Jones and Sons Co. of Pittsfield, Mass. It is located in the basement of the building under the final calender stacks and reel and replaces a former conventional broke beater.

Illustrative of the most up-to-date equipment included in the modernization program is the beta ray gage added to the mill's no. 1 paper machine. This provides continuous measurement of the weight of the paper sheet as it is produced on the machine. Utilizing radioactive material provided by Industrial Nucleonics Corp., a sealed capsule of radioisotopes traverses the width of the paper web just prior to its being rolled on reels. In addition to making possible extremely accurate basis weight control, the equipment benefits production by permitting faster startups and changes and reduces waste by eliminating the necessity to tear out samples as was previously required.

New equipment and improved processing were included in the program for the pulp mill and finishing operations in order to utilize the greater capacity and capabilities of the new paper machine. Pulp washing in the bleach plant, which previously had been one of the limiting factors in production, was stepped up by replacement of older inadequate units by two 8 by 10 ft. vacuum washers produced by the Improved Machinery Co. of Nashua, N. H.

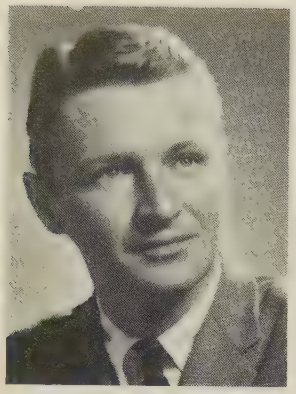
More effective storage and increased handling capacity through more efficient high-density tank operation were achieved by the addition of agitation equipment and mining nozzles supplied by the James Brinkley Co. of Seattle. Magnetic flow meters from Foxboro Instrument Co. and consistency regulators supplied by the DeZurick Corp. for measuring and controlllng pulp delivery have been incorporated in the high-density storage system and at the deinking plant output.

The company's deinking plant is a considerable factor in Simpson's conservation program which stresses greater and more intensive utilization of raw materials, especially of those which were formerly considered as waste or of no commercial value. Additional pulp capacity was achieved by installing two slushmakers produced by the Morden Machines Co. of Portland and enlarging and adding to associated washing equipment. These improvements have more than doubled the capacity of that section of the pulp mill and permit the input of 70 baled-weight tons of magazines per day as compared with the former 33 tons daily.

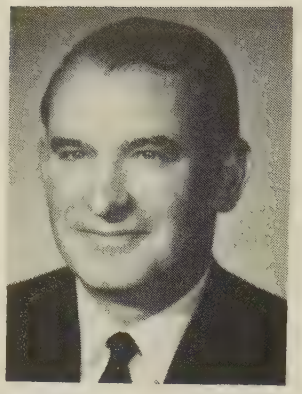
Three years ago Simpson engineers undertook a project to develop a facility for the continuous production of calcium hypochlorite used in the bleaching stages of pulp manufacturing. Utilizing equipment originally furnished by Pennsylvania Salt Manufacturing Co. of Tacoma, Simpson personnel redesigned and increased the capacity to manufacture the bleach as needed. The system, just completed last month, mixes chlorine in proper proportions to a lime slurry of appropriate consistency to produce the calcium hypochlorite in quantities sufficient to meet the needs of the pulp plant without the necessity for additional storage tanks with their attendant settling and clarifying requirement.

About the same time, action was initiated to prepare chip production and handling facilities to cope with the anticipated greater demand which a new and faster machine would create. The W. H. Rambo Industrial Plan Engineers of Portland were called upon to design a larger debarking plant to prepare logs and wood for the chipping operation. With construction and installation under the direction of the H. S. Wright Co., a new plant was completed in November, 1956, capable of feeding 40 ft. logs into a 50 in. mechanical ring barker supplied by the Nicholson Manufacturing Co. of Auburn, Wash. Log haul facilities and conveyor systems supplying the barker and delivering debarked logs to splitters and chippers were fabricated by the American Boiler Works of Everett.

Improvements were also effected on the mill's no. 2 paper machine prior to the installation of the new Royal Chinook. About two years ago a coating-size press was added to the older machine and an entirely new afterdrying section consisting of seven additional 4-ft. drying cylinders installed to add to the versatility of grades which could be produced on that machine. The size press was manufactured by Rice-



W. R. Denman, Jr., Simpson Lee Paper Co.



Derek Currie, Weyerhaeuser Timber Co.

Barton of Worcester, Mass., and Black-Clawson of Hamilton, Ohio, supplied the additional driers. At the same time a 440-hp General Electric turbine was installed for the main drive and a Morden pulper added for broke reclamation. Since the startup of the Royal Chinook, a Pope-type reel produced by the Pusey Jones Corp., Wilmington, Del., has been added to the no. 2 paper machine permitting the handling of larger rolls.

The greater papermaking capacity of the plant required enlargement of finishing and shipping facilities. These were also anticipated by the addition of new equipment and provisions for streamlined automatic handling of the paper as it flowed from the paper machines into the new finishing room, converting plant, and warehouse which had been constructed in 1955. Two years ago an automatic high-speed rotary cutter 100 in. wide and with automatic layboy capable of handling 60 tons of paper a day was installed. Two other cutters have had automatic layboys added.

Originally two highly mechanized trimmer lines were installed in the finishing department. Just recently a third automated trimmer line was added in that department and three such lines included in the converting department for the production of small cut size reams. These three automated lines are integrated and flexible so any combination can be fed into two automatic wrappers.

The trimmers in both the finishing and converting departments are provided with sheet metal ducts carrying a high velocity stream of air which provides a pneumatic removal of the trim waste or broke to a centralized collection point where the broke can be transformed again into pulp and reintroduced into the papermaking cycle. Such a system minimizes waste and reduces labor and handling costs.

The installation of the Royal Chinook and completion of modernization programs attendant thereto have increased the plant's average production to 140 tons of paper daily. When the old no. 3 paper machine was installed in 1926, the plant's capacity was doubled from 35 to 70 tons daily. Completion of further modernization six years ago raised this capacity to over 100 tons daily. The recent programs described herein accomplished the greatest single production increase since the original third paper machine was installed, and provide the basis for even greater future capacity and expansion when warranted by greater paper usage and growth in demand.

The promotion of W. R. Denman, Jr., to sales manager for printing and fine papers has been announced.

Formerly administrative assistant for sales, Denman has been associated with the West Coast pulp and paper manufacturing concern for seven years. A graduate of the University of Washington, he started his career with Simpson as a laboratory technician, later becoming a salesman covering the Northwest territory out of the company's Seattle sales office. He returned to the Everett mill in 1954 when the quality control department was established and was its supervisor at the time he was selected to assist the general manager in matters pertaining to sales.

Weyerhaeuser

Howard W. Morgan, vice-president, pulp, paperboard, and container group of the Weyerhaeuser Timber Co., announced the appointment of Derek Currie as manager of the Pulp Division of the Company, a position formerly held by Mr. Morgan. As manager of the division, Mr. Currie will be responsible for all production, sales, engineering, and research for the division.

Mr. Currie was formerly vice-president and general manager of Eastern Fine Paper & Pulp Division of Standard Packaging Corp., Bangor, Me. He was born and educated in Britain where he also received his early training in the paper industry. After five years of military service, he was appointed director of paper in the Indian government, being responsible for the control and coordination of the production of the Indian paper industry.

Mr. Currie joined the E. B. Eddy Company, Hull, Canada, in 1946 where he took an active part in the integration and modernization of the Eddy and Booth mills. He became manager of manufacturing in 1950 and was subsequently appointed vice-president of manufacturing and a director of the company. In 1957 he joined the Eastern Corp., Bangor, Me., as vice-president and assistant to the president and became general manager in 1958.

Consolidated Water Power

G. Kent Dickerman, Jr., has joined the Appleton Div. of Consolidated Water Power & Paper Co. He will serve as a chemist in the division's technical department.

Mr. Dickerman was graduated from Western Michigan College with a B.S. degree in chemistry and has been employed as a chemist by Minnesota Mining & Manufacturing Co. since 1956.

Rayonier

S. Earll Church has been appointed manager of Rayonier's technical service, James T. Sheehy, executive vice-president, has announced. He replaces George B. Creamer who was earlier named assistant general manager of research.

Mr. Church, who joined Rayonier as a technical service representative two years ago, had been associate professor of pulp and paper technology at State University of New York at Syracuse. Technical service, a special Rayonier agency established under the cellulose producer's sales organization, assists world cellulose-users in the application of Rayonier pulps to their specific needs.

KVP

Robert D. Caine, vice-president in charge of manufacturing for the KVP Co., Kalamazoo, announces the appointment of Richard J. Moser as assistant superintendent of the papermaking division at the company's Kalamazoo plant.

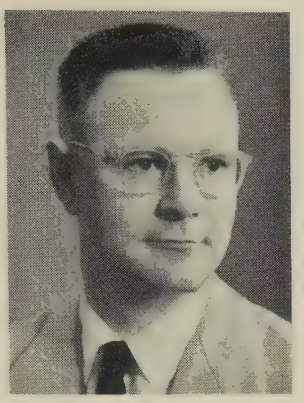
Mr. Moser joined KVP on June 9, 1952, immediately following receipt of a B.S. degree in chemical engineering from Michigan State University.

Mr. Moser's first assignments with the company were in the Kalamazoo plant's research laboratory and papermaking division. In 1957, he was transferred for one year to the company's pulp and paper mill in Espanola, Ont., Canada. Since returning to the Kalamazoo plant, Moser has been assigned as a staff assistant to the wax and printing divisions.

The KVP Co., Ltd., has approved plans for a 15,000 ton per year expansion of specialty paper production at its mill in Espanola, Ont., A contract for supplying the major equipment has been awarded to Black-Clawson (Canada) Ltd., Montreal.

Decisions concerning the design details for much of the equipment were made recently at meetings in England between the engineering staff of the KVP Co. and officials of

The Black-Clawson Co. from New York, Montreal, and London. Manufacture of the equipment will be shared by Black-Clawson facilities in Canada, United Kingdom, and the United States.



R. J. Moser, KVP Co.

The project calls for a new 136-in. combination Yankee paper machine, a complete stock preparation system, and a Flexiblade off-machine coating unit.

The paper machine will be designed for production of machine glazed and machine finished papers, particularly in the bread wrapping and waxing field.

The expansion program is designed to maintain the established high quality of production in this mill as well as increase its paper capacity nearly 50%. The new Black-Clawson paper machine will be the third machine in this mill.

BUCKEYE

Buckeye Cellulose Corp. has doubled the original capacity of its five-year-old wood pulp plant at Foley, Fla., with the completion of a new $20 million mill. The new capacity is 200,000 tons of dissolving pulp per year, or 266,000 tons of paper pulp.

The new Foley mill is integrated with the original mill in a semi-independent tandem arrangement. Both are linked department by department, but either can be operated independently of the other at all times, if desirable. Both mills can run one grade of pulp if orders demand, or can run different pulps to accommodate the schedules of more customers. This flexibility was a major consideration in the design and engineering of the new mill, in order to gear it as much as possible to customers' requirements.

Among the notable features of the new mill is a unique double screening system for the removal of undesirable particles prior to bleaching. With the new system every fiber passes through at least two sets of screens before bleaching treatment. Initial trial runs showed the new system to be so effective that the same equipment was installed in the original mill.

Other unusual features include:

A special seven-stage bleaching process developed by Buckeye researchers and employed in both the new and the old mill.

An unusually high efficiency rate of more than 90% in the recovery and re-use of pulping chemicals. In normal operation, the recovery rate is expected to exceed 95%.

Completed early in 1954, the original Foley mill started production in June of that year with a capacity of 100,000 tons and represented a capital investment of about $30 million. An interim expansion completed in July, 1956, raised capacity to 120,000 tons a year.

The newly completed mill, boosting capacity to 200,000 tons a year, brings the total capital investment at Foley to $50 million.

Buckeye, the only company in the industry producing dissolving pulp from both wood and cotton linters, also operates a linter plant in Memphis, Tenn., with an annual capacity of 115,000 tons. The Foley and Memphis units give Buckeye a total capacity in excess of 315,000 tons per year.

Within the last six years, Buckeye's total capacity has tripled, making it the fastest-growing producer in the industry.

Structural

A total of approximately 2100 tons of structural steel was erected, and approximately 1700 tons of piling were driven.

Instrumentation

Generally, Foxboro instruments were used in the process departments. Remote flow recorders, some temperature recorders, gas analyzers, computing relays and manual loading stations were by Bailey Meter Co., who supplied the instrumentation for the recovery boiler. Multipoint temperature recorders were by Honeywell. Pneumatic temperature transmitters were by Moore Products. Standard control valves were De Zurick with Bailey positioners, and consistency transmitters were De Zurick in-line type. Butterfly valves were supplied by Continental Equipment Co.

Woodyard

With daily wood requirements increased more than 86%, additional wood storage facilities for 11,000 cords adjacent to, and for 5000 cords within, the existing woodyard have been provided for the expanded operation. A new log conveyor from the new area loads a new barking drum (Fibre Making Processes, Inc.), from which the logs are conveyed to a new sorting belt on the extended sorting floor.

A new hardwood barker (Impco), with new infeed conveyor and outfeed conveyor discharging to the existing sorting belt, will be started when volume hardwood operations begin.

Basically, existing facilities for log recycle, cull hogging, log splitting, chipping, chip screening, chip conveying and storage, handle the entire flow. Conveyors were speeded up, one existing chipper was converted, and a new chip silo was erected in order to accomplish this.

A new bark hog (Diamond Iron Works) was added to handle the increased bark load. Surplus bark will be conveyed pneumatically to a new conical type refuse burner (Rees Burner and Blowpipe).

New bulk chip handllng facilities include: an hydraulic truck dumper (Kewaunee), a rail car shaker (Link-Belt), a receiving pit, a takeaway conveyor, and a 7800 sq. ft. concrete storage mat.

The existing chip feeder conveyor under the chip silos was extended to pick up the new silo, and the existing conveyors carrying chips to digesting were speeded up to handle the higher load. The digester charging conveyor was extended to include the five new digesters. The existing off type tripper was moved over the new digesters, and a new feed-through or off-type tripper was installed over the existing digesters. All new conveyors, conveyor conversions, and the new tripper are by Continental Gin Co.

Digesting

Expanded operation of the digesting department is provided for by the addition of five new 4150 cu. ft. capacity stainless clad digesters (Chicago Bridge & Iron Co.). Each digester is provided with an indirect solution heater (Downington Iron Works) and a new Goulds solution circulating pump.

One digester is arranged for swing operation and the other four are permanently piped to the new two-charge blow tank and new entrainment separator (Hammond Iron Works). Associated with the blow tank is a heat accumulator tank with an internal direct contact condenser (Chattanooga Boiler and Tank Co.) and an accumulator condenser water pump (Goulds). Operating in cooperation with the heat accumulator tank are two heat exchangers (Struthers-Wells) and a dirty water pump (Goulds).

Two notable features of the new digester department are the integrated heat revovery system, made possible by special instrumentation, and the automatic cooking solution mixing system.

Operation of the integrated heat recovery system is made possible by the special instrumentation. Other than that already mentioned, the only additional equipment is a Goulds hot water pump and a Goulds fresh water booster pump.

The new solution mixing tank was built by Chattanooga Boiler and Tank Co.

Washing and Screening

The new washing and screening department is essentially a duplicate of the existing department except for changes in suppliers of some of the equipment.

The new Sandy Hill Kamyr knotter stock pump is V-belt driven through a dynamatic variable speed coupling and is controlled by a new Foxboro magnetic type flow controller. Three new Bird Machine Co. Jonsson knotter screens are installed ahead of a new three stage Impco brownstock washing system. The associated liquor storage tanks and foam tower were built by Chattanooga Boiler and Tank Co. The various liquor pumps, and the white water pump, were supplied by Goulds. The three filtrate pumps are powered by 250 hp., 2200 volt grade mounted weather protected Allis-Chalmers motors.

The new screening section is composed of six primary and two secondary Impco Lindblad vibrating screens, one Impco flat screen and four Impco centrifugal screens. The new Impco valveless decker, which is driven through a dynamatic variable speed coupling discharges stock to an Impco thick stock pump, which pumps to two 50-ton high density storage towers built by Chattanooga Boiler and Tank Co. The tile white water chest and primary screen stock mix tank were built by Chemical Linings, Inc. Stock from the high density towers is pumped to an agitated blend chest, built by Chemical Linings, Inc., where consistency of stock to bleaching is adjusted.

Bleaching

The new mill features a seven-stage bleach plant, with major equipment supplied by Impco. Eight tile-lined steel bleach towers consist of two low density upflow chlorine towers in series and six high density downflow towers.

Tanks were bullt by Graver, and tile lined by Chemical Linings, Inc. Impco supplied all launders, circulators, and mining nozzles. On the bleach floor are seven Impco 9 ft. 6 in. diam. by 14 ft. stainless steel washers. Washer seal boxes were built by Chemical Linings, Inc., and all dilution pumps were supplied by Goulds. Stock to bleaching passes through two rubber-lined Impco chlorine mixers. and stock from the washers passes through five Impco 500-ton stainless steel steam mixers, two Impco hastelloy C chlorine dioxide mixers, and two Impco 500-ton thick stock pumps.

All other stock pumps were supplied by Sandy Hill or by Allis-Chalmers. All high density tower mining water pumps were supplied by Buffalo. Stock from the bleach plant is stored in two 42 ft. diam. by 43 ft. high tile Impco low density stock storage chests built by Chemical Linings, Inc. Circulators were supplied by Impco. All motor control center, washer hood, and comfort ventilating systems are J. O. Ross. All major process piping is either stainless steel or lined carbon steel.

Pulp Drying

Stock preparation for the new pulp drying department consists of two 36 in. Bauer Bros. Magna Cleaners, followed by a conventional vortrap cleaning system (Nichols-Freeman). As in the original mill, stock is thickened over a valveless decker (Sandy Hill). The tile stock chest and white water chest were built by Chemical Linings, Inc.

The pulp drying machine (Sandy Hill) is 180 in. wide, with a Bertrams flow distributer supplying a 100 ft. long conventional fourdrinier type wet end, followed by a suction couch first suction wet press, four-drum predrier section, second suction wet press, and a smoothing press. The drying section consists of a 48-drum first stage, a breaker stack, and a 13-drum second stage, followed by a three-drum suction air cooler of new design (Sandy Hill), draw rolls, and reel. The machine is driven by a Sandy Hill selective line shaft drive powered by a Westinghouse 400-hp. d.c. motor. The machine hood and all air systems were supplied by J. O. Ross. Notable among the machine auxiliaries is the fourdrinier vacuum system which consists of four Nash Hytor vacuum pumps driven in tandem by an Allis-Chalmers 1000 hp. 360 r.p.m. 2300 v. synchronous motor.

To accommodate the increased drying capacity, additional storage facilities for 1400 tons of jumbo rolls has been provided.

Warehouse space has been increased by 80,000 sq. ft. of storage area, plus 2400 sq. ft. of loading dock. Loading facilities for 8 boxcars—4 all weather and 4 outside—have also been added.

Finishing

The new finishing equipment consists of Lambs-Gray Harbor Co. backstands, cutter and layboy, roll rewinder, and bale conveyors. The new 2600 ton hydraulic press was supplied by Washington Iron Works and utilizes Tennant automatic Twist Lock wire tying machines. Conveyors can be set for continuous operation with bales fed from the typing machines directly to the bale stacker, or they can be set to accumulate bales and allow intermittent operation of the bale stacker. Another feature of the combined system is that cut sheets from no. 1 layboy can go to the no. 2 finishing line or vice versa.

Causticizing

The new causticizing department, while generally similar to the existing, contains some notable departures from it. The new oil-fired Allis-Chalmers kiln is located alongside the existing unit which it duplicates, and is served by extensions from the old feed and firing houses. Notable features of the kiln installation are the two Bird Machine Co. lime slurry centrifuges used in place of the original cylindrical vacuum filters.

One of the machines is used to dewater sludge from the cold lime softener, allowing recovery of lime ordinarily lost to the sewer, for reuse in the cold lime softener. Another feature of the new kiln is the variable speed induced draft fan driven through a Dynamatic coupling which is remotely adjusted from the firing panel to maintain proper combustion conditions continuously monitored by a Bailey oxygen analyzer.

Other

Other major equipment added is as follows: green liquor and white liquor storage tanks built by Graver; green liquor indirect heater-cooler by Downingtown Iron Works which differs from the original direct contact heater, lime slaker-classifier built by Dorr-Oliver; three new stainless lined causticizing tanks built by Hammond Iron Works with Dorr-Oliver agitators and field-applied lining; white liquor clarifier and lime mud washer both built by Hammond Iron Works with Dorr-Oliver internals; lime and storage tank built by Hammond Iron Works with Dorr-Oliver agitator; and water treat sludge storage tank built by Hammond Iron Works with Mixco agitator and recarbonation injection piping. All diaphragm sludge pumps are by Dorr-Oliver. Centrifugal pumps were supplied by Goulds.

Another new system is the Davenport Machine and Foundry air cooler and the pneumatic conveyor to cool and transport reburned lime to the water treatment lime bins. The conveyor utilizes the existing Fuller lime unloading system for its operation.

Evaporation

The new evaporation department is a five effect unit supplied by Chicago Bridge and Iron. All pumps are by Worthington.

Steam

Steam requirements for the expansion have been provided

for by the addition of a new 550-ton Babcock and Wilcox chemical recovery boiler. Actual steam capacity is 285,500 lb. per hr. at 600 p.s.i. and 750° F.T.T.

Notable among the features of the new boiler is the Venturi evaporator-scrubber system which takes the place of the cascade evaporators and the electrostatic precipitator which were used on the original recovery boiler. The unusually powerful induced draft fan necessitated by the 38-in. draft loss across the Venturi scrubber system is an American Blower 835 r.p.m. compound type with all rotating parts of stainless steel and with Corten housing. The fan is driven by 2700 hp. 5000 r.p.m. General Electric back pressure type steam turbine. The horizontal economizer and vertical gas air heater are provided with a shot cleaning system. The original recovery boiler was not provided with a gas air heater. The new stack is 225 ft. brick lined concrete with salt cake storage facilities in the base, and was designed and erected by Rust Engineering Co.

The exhaust steam from the 10,000-k.w. turbogenerator and from the induced draft fan turbine supplies the new 50 p.s.i. header which serves the new pulp drying department, and ties into the old 50 p.s.i. header serving the original pulp drying department and both bleach plants. The new header has steam make-up through a new pressure reducing and desuperheating station. Two additional new pressure reducing and desuperheating stations supply make-up for the 50 p.s.i. header to the new black liquor evaporators and for the extended 150 p.s.i. digester steam header.

Power

Power requirements for the expansion have been provided for the addition of a 10,000-k.w. General Electric noncondensing turbine driven generator. Capacity of the purchased power tie is unchanged. Total plant generating capacity is now 26,600 k.w. at 85% P.F.

Power distribution for the new equipment has been provided by installing two new, and extending four existing, 12.47-k.v. feeders for a total of nine in the entire plant. Fifteen new Allis-Chalmers substations have been installed, adding a connected capacity of 15,000 k.v.a., for a total of 35 substations with a total connected capacity of 35,500 k.v.a. Twelve of the new substations are 480-v. secondary type for low voltage motor control, lighting, instrumentation, welding supply, elevators, and cranes. Three of the new substations are 2300-v. secondary type for control of motors larger than 200 hp. All but three of the new substations are located indoors in ventilated control rooms.

Power for the new wells is provided by a 12.47-k.v. pole line and individual banks of 100-k.v.a. transformers.

Motor control centers are Square D and all but five are located indoors in ventilated control rooms. Three are outdoors, and two are indoors in operating areas.

Motors are generally squirrel cage induction type, of totally enclosed chemical construction. Motors 200 hp. and below are 480 v. and were supplied by Westinghouse. Those 250 hp. and above are 2300 v. and were supplied by Allis-Chalmers. The notable variations in motor type and construction are the paper machine drive motor, vacuum pump drive motor, main mill water pump drive motors, and liquor pump drive motors in the brownstock washing system.

Water

Water requirements for the expansion have been provided for by extensive additions to the water supply and treatment facilities. Three new wells with a combined capacity of 26 million gal. per day were drilled by Layne-Atlantic, who also supplied the pumps. The new well line is composed of 10,000 ft. of 42 in. and 2500 ft. of 36 in. Lock Joint 150 p.s.i. prestressed cylinder type reinforced concrete pipe, and is sized to permit the later addition of two wells.

The original no. 1 well, which is located inside the plant property itself, has been abandoned as a full capacity well and was converted for use as the potable water supply source.

A new 125-ft. diam. wirewound prestressed concrete cold lime softener was designed and erected by the Crom Corp. and essentially duplicates the original. Softener internals are by Graver. The softener is provided with a wood cover and has automatic desludging controls. New chemical feeding equipment was installed. Included are three pneumatically paced Omega continuous weighing feeders. Two feeders replace existing Omega intermittent type units over existing slakers and one was provided with a new slaker to serve as a swing unit.

Also added were two Builders Iron Foundry chlorinators for the treatment of all water flow through both softeners, and one new Wallace & Tiernan silactor to serve the new softener. Water from the new softener passes through two American Well Works in line type recarbonators duplicating the original.

A new 2 million gal. wirewound prestressed concrete reservoir with integral concrete shell dome was designed and erected by The Crom Corp. and duplicates the original. Two new Goulds 10,000 g.p.m. 150 ft. head main mill pumps, powered by two 500-hp. Allis-Chalmers grade mounted weather protected motors, supply general service water through a new 36 in. cast iron and carbon steel water supply line. Two new Allis-Chalmers filter supply pumps powered by Westinghouse TEFC motors supply additional water to the expanded ion exchange plant through a new 14 in. cast iron header. Nine new 10 ft. diam. Belco sand and gravel pressure filters, one new 10 ft. diam. Graver acid cation exchange unit, three new 10 ft. diam. Graver sodium cation exchange units, and two new 10 ft. diam. Graver anion exchange units were added to gain the necessary capacity increase in the ion exchange plant.

In addition to these a new sodium backwash sump with controls was installed so that it is possible to regenerate one of each type ion exchanger simultaneously. Soft water for the expanded plant is handled through a new tile storage chest, soft water pump, and soft water header. A new boiler feedwater pump was added at the existing demineralized water storage chest.

Air

Increased factory compressed air requirements were provided for by the addition of a new 100 p.s.i. 450 c.f.m. Worthington horizontal single cylinder, single-stage, double acting air compressor, driven by a 100-hp. motor.

Instruments for the new boiler and black liquor evaporators were provided for by the addition of a 90 p.s.i. 180 c.f.m. Worthington horizontal single cylinder, single-stage, double acting air compressor driven by a 40-hp. Worthington steam turbine. Increased process instrument air was provided by the addition of two 100 p.s.i. 160 c.f.m. Gardner-Denver single cylinder, single-stage, double acting nonlubricated carbon ring type air compressors driven by 40-hp. motors.

Newton Falls

George S. Carroll and his wife Althea were recently honored at a farewell party at Newton Falls, N. Y. Mr. Carroll is retiring from active service in the paper industry after 45 years with the industry. The last 11 years have been spent in the employ of the Newton Falls Paper Co., as general superintendent and assistant to the president. Previously, Mr. Carroll was associated with the American Writing Paper Co., Monadnock Paper Mills, and the Hopper Paper Co.

Mr. Carroll plans to spend his summers at his home in East Washington, N. H., and his winters in Orange Lake Village, Largo, Fla., where he has recently purchased a home. He plans to be available for consulting on a limited basis.

Hammermill

Donald T. Jackson has been elected a vice-president and William Beckett a director of the Hammermill Paper Co., it was announced recently by President Donald S. Leslie following a board meeting.

Mr. Jackson has been Hammermill's technical director since 1954 and will continue to head the Technical Department as a vice-president. As such, he will be responsible for research and development, laboratory and engineering work not only at the Erie mill, but at all Hammermill divisions and subsidiaries.

Currently he is directing the over-all phases of the Erie mill's $20,000,000 growth program, a three-year program which is now nearing completion. In addition, he headed up the research work that led to the development several years ago of Hammermill's Neutracel pulping process.

An industrial chemistry graduate of Montana State College, Mr. Jackson received his Ph.D. degree from the University of Pittsburgh in 1932. He taught chemistry for several years at both institutions.

In 1933, he joined Hammermill's laboratories as a chemist. The next year he was appointed assistant chief of the research laboratories, the first in a series of promotions which saw him move up to assistant director of the laboratories in 1945, director of the laboratories in 1948, director of research and development in 1953, and technical director in 1954.

Mr. Jackson is a member of the American Chemical Society, TAPPI, and is chairman of the Pennsylvania Section of the Operating Committee for the National Council for Stream Improvement. He is a recognized authority on pulping, pulp bleaching, waste recovery and treatment—and has written numerous technical articles which have appeared in various technical and paper trade publications.

William Beckett, new member of Hammermill's board of directors, is president of The Beckett Paper Co., Hammermill's recently acquired wholly owned subsidiary.

A lifelong resident of Hamilton, Ohio, Mr. Beckett has long been active in political and community affairs, as well as the paper business, in his native city. He has served as a city councilman for six terms, mayor of Hamilton for two terms, and recently completed a term as state senator in the Buckeye State.

Mr. Beckett attended Massachusetts Institute of Technology where he majored in chemical engineering. He has been associated with the 111-year-old company his grandfather founded for the past 26 years, working successively in manufacturing, quality control, order department, and as office manager, treasurer, executive vice president, and, since 1958, president.

During both World War II and the Korean War, he served as an intelligence officer with the rank of major in the Strategic Air Command.

In the paper industry, he is a former president of the Pulp Consumers' Association and is a member of TAPPI.

In addition to his directorship at Hammermill, Mr. Beckett is a member of the board of directors of the Cincinnati Gas and Electric Co., Crystal Tissue Co., and the Citizens Bank in Hamilton.

Continental Can

Indications of a possible economic upturn for the pulp and paper industry were evidenced with the report that The Babcock & Wilcox Co. has received a major contract to manufacture and install steam generating equipment at the new paper mill of Continental Can Co.'s Robert Gain division in Nixon, Ga.

Under terms of the contract, valued at more than $2 million, Babcock & Wilcox will furnish two boilers, a combination power and bark-burning unit, and a kraft-type black liquor recovery boiler, for the huge bleached sulphate paperboard and paper mill.

Babcock & Wilcox officials attached great significance to the contract. "We believe it signals the beginning of a new round of mill expansions throughout the pulp and paper industry after two years of almost complete inactivity," they said.

W. L. Schnorbach, Continental Can Co., Inc.

Scheduled to go into operation in late 1960, the mill will have an initial capacity of 350 tons a day. Paper industry sources estimate that the plant, located on a 2600-acre tract 11 miles south of Augusta, Ga., along the Savannah River, will cost nearly $40 million when completed.

Designed for a capacity of 400,000 lb. of steam per hr. with a two-hour peak capacity of 480,000 lb. the power boiler will operate at a pressure of 875 p.s.i. and a temperature of 900°F. at the superheater outlet. Approximately one third of the boiler's capacity will result from bark firing at a rate of 40,000 lb. per hr. A feature of the unit is that to meet the mill's steam demands, the bark firing can be supplemented by the burning of natural gas at a rate of 406,000 cu. ft. per hr.

The recovery unit, designed for a capacity of 185,000 lb. of steam per hr., will operate at the same conditions as the power boiler. It is sized to process black liquor solids resulting from the production of 400 tons of bleached board per day. Bleached board includes such products as frozen food packaging materials, milk and ice cream carton board, and plates, cups and containers for similar application.

J. E. Sirrine Co., of Greenville, S. C., is the consulting engineer for the construction of the new mill.

William L. Schnorbach has been appointed plant manager of Continental Can Co.'s new bleached board mill now under construction at Augusta, Ga., it has been announced by C. B. Stauffacher, executive vice-president of the firm's Robert Gair Paper Products Group.

Mr. Schnorbach had previously been manager of special process and project engineering with the Gair Group, working on plans for construction of the mill. He had joined the Robert Gair Co. (which merged with Continental Can in 1956) in May 1955, as special representative on containerboard production, progressing from there to become assistant to the director of development, chemical pulp and paper, and then to his process and project engineering assignments.

A graduate of the University of Michigan with a degree in chemical engineering, Mr. Schnorbach has spent his entire business life in the paper industry.

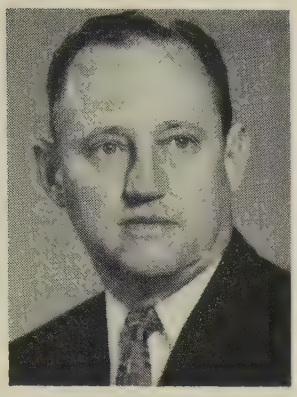
The new bleached board mill which he is to manage will produce 350 tons per day of bleached paper and board for use in packaging food products. The mill is expected to be in operation during the last quarter of 1960 with the initial 350-ton capacity to be provided by a 216-in. trim fourdrinier paper machine.

The former manufacturing plant and sales office of the Fort Wayne Corrugated Paper Co., located at 4500 South Kolin Ave., Chicago 32, Ill., have been added to the Fibre Drum and Corrugated Box Division of Continental Can Co., it was announced by P. Wojtul, Division vice-president.

Continuing to supervise this operation will be H. R. Chaney, plant manager, T. E. Kigin, district sales manager, and A. F. Nichols, assistant district sales manager.

The Fort Wayne Corrugated Paper Co. merged with Continental Can Co. on June 15. Continental sold the other three of Fort Wayne's four corrugated box plants which were located in Hartford City, Ind., Rochester, N. Y., and Pittsburgh, Pa.

G. L. Bidwell, Riegel Paper Co.



C. E. Hartford, Riegel Paper Co.

R. R. Morris, Crown Zellerbach Corp.

W. O. Beaton, Crown Zellerbach Corp.

RIEGEL

F. S. Leinbach, president of Riegel Paper Corp., has announced the following corporate changes.

A. P. Mitchell, senior vice-president-sales, has been appointed to a newly created post, director of marketing. He will continue as senior vice-president.

The name of the New Jersey Division has been changed to the Specialty Products Division in order to describe more completely the nature of its operations. G. Lamont Bidwell, formerly vice-president and manager, has been named vice-president and general manager. Charles E. Schaehrer has been elected a vice-president of the corporation, and has been named sales manager of the Specialty Products Division, reporting to Mr. Bidwell. He will also continue his present duties as manager, packaging materials sales. A. J. Ericsson, product manager for merchant and industrial sales, O. E. Lohrke, manager of merchant sales, and Benjamin Foose, Jr., manager of industrial sales, will report to Mr. Schaehrer in his new capacity.

In the Carolina Division, C. E. Hartford has been named vice-president and general manager. He was formerly vice-president and manager. Winthrop Endicott has been named sales manager of paper and paperboard for the Carolina Division reporting to Mr. Hartford. He was formerly manager, merchant and industrial sales.

The Quality Lithographing Division will continue under the direction of Chalmers W. Barr, general manager.

G. Lamont Bidwell, vice-president and general manager of the Specialty Products Division, Riegel Paper Corp., has also announced the following promotions.

W. A. Schenck, formerly Upper Mills manager, has been advanced to the newly created position of manager of paper production, reporting to Mr. Bidwell. In this capacity, Mr. Schenck will be responsible for all paper machine production and finishing in the Warren Glen, Hughesville, Riegelsville, and Milford Mills.

L. A. Moss, formerly production manager of the Milford Mill department, has been named manager of manufacturing services reporting to Mr. Bidwell. In this capacity he will be responsible for engineering, laboratory, quality control, industrial engineering, purchasing, traffic, production control, and maintenance planning.

C-Z

Crown Zellerbach Corp. has announced two promotionsi n its Northwest mill operations. R. R. Morris, resident manager of the corporation's Lebanon, Oreg., mill, has been promoted to assistant resident manager of the Camas, Wash., mill, and W. O. Beaton, technical assistant to the paper mill superintendent at Camas, succeeds him as resident manager at Lebanon.

A graduate of the University of Washington, B.S. in chemistry, Mr. Morris joined Crown Zellerbach in 1950 as a chemical engineer at Camas. He was later promoted to technical assistant to the paper mill superintendent and, in April, 1957, was transferred to Lebanon as resident manager.

Mr. Beaton is a mechanical engineering graduate of the University of Washington. He joined the corporation in 1952 as a draftsman at the Port Townsend mill, where he was soon promoted to technical assistant, multiwall bags. In 1953 he was transferred to Camas as assistant to the superintendent, multiwall bags and, in 1957, he became bag factory superintendent there. He was promoted to technical assistant to the paper mill superintendent at Camas in August, 1958.

A series of management changes in Crown Zellerbach Corp.'s Gaylord and Western-Waxide packaging divisions have been announced in San Francisco by David J. Benjamin, vice-president for packaging.

Lloyd Merwin, Sr., of St. Louis, Crown Zellerbach vice-president, remains general manager in charge of the Gaylord division. In this position, Mr. Merwin is responsible for the division's 17 corrugated shipping container plants and 62 sales offices throughout the United States.

Three newly appointed corporation assistant vice-presidents in the Gaylord division's St. Louis executive offices will report to Mr. Merwin.

W. G. Hunt, formerly manager of converting plant engineering and development, was named assistant vice-president for manufacturing and engineering.

Edwin J. Spiegel, Jr., who has been assistant to the vice-president and general manager, was appointed assistant vice-president for administration for the Gaylord division.

M. M. Jamieson, general sales manager for the shipping container division, was named assistant vice-president for sales.

In the Western-Waxide division, a nation-wide converter and distributor of flexible and specialty packaging, M. A. Barr, assistant general manager for production, was appointed divisional general manager, with headquarters in San Leandro, Calif.

R. L. Appling, formerly assistant general manager for sales, was named assistant general manager and will continue to be in charge of sales.

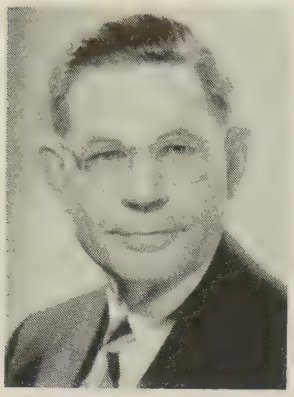
E. A. Mitchell, former assistant general manager for administration in San Leandro, becomes an assistant vice-president for packaging in San Francisco.

I. P.

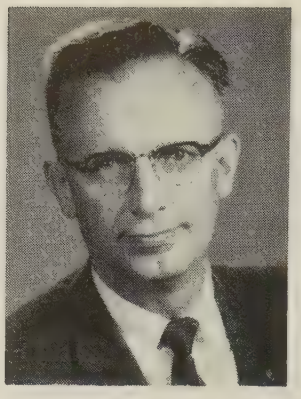
International Paper Co.'s Container Division has purchased property and is building a new corrugated box plant at Phoenix, Ariz., it was announced July 28 by Arthur B. Damon, division general manager.

The plant is scheduled to be in operation within a few months.

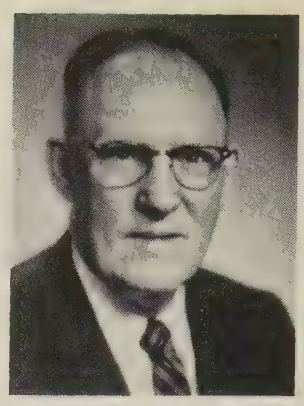
Mr. Damon also announced the appointment of Donald M. Scott as sales manager for the new operation. Mr.



G. H. Rand, International Paper Co.



R. C. Masterman, International Paper Co.



H. C. Nelson, Owens-Illinois Glass Co.

F. C Keeney, Knowlton Bros.

Scott, a native of Los Angeles and a graduate of the University of Southern California, has been with the International Paper Container Division since 1953 as a salesman in the Los Angeles metropolitan area.

The promotion of George H. Rand to the executive staff of International Paper Co. as assistant to Stuart E. Kay, vice-president, has been announced by Richard C. Doane, president.

Mr. Rand, who has served as divisional manager for the Northern mills since 1954, will move to the company's New York offices where he will assist Mr. Kay on operations of the Northern Division and on special assignments.

A native of Maine and a graduate of Bowdoin College, Mr. Rand joined International Paper in 1930. He served at the company's mills in Livermore Falls, Me., and Niagara Falls, N. Y., before being transferred in 1952 to divisional headquarters in Glens Falls, N. Y., as assistant divisional manager.

Mr. Doane also announced that Roscoe C. Masterman has been appointed to succeed Mr. Rand as divisional manager for the Northern mills.

Mr. Masterman, also a native of Maine, graduated from the University of Maine before joining the company in 1933. He was employed at the Otis Mill in Livermore Falls from 1933 to 1956 when he transferred to Glens Falls as assistant divisional manager.

Mills coming under Mr. Masterman's supervision include the Otis Mill, mills in Niagara Falls, Tonawanda, Ticonderoga, and Corinth in New York and a kraft specialty mill at York Haven, Pa.

Peninsular

After 47 years with the Peninsular Paper Co. of Ypsilanti, Mich., Mill Superintendent Joseph J. Ely has announced his retirement. Mr. Ely started with Peninsular as a machine room worker in 1912. He became foreman in 1932 and has been superintendent for the past 23 years.

A unique aspect of Mr. Ely's retirement is that he has been succeeded as superintendent by his son, John Ely. John is a 25-year employee of Peninsular, and has been assistant superintendent since 1951.

O-I

Henry G. Nelson, quality supervisor of the Shipping Container Research Laboratory of the Glass Container Div. of Owens-Illinois Glass Co., has been named coordinator of quality control for the company's Paper Products Div.

Mr. Nelson's promotion was announced by John. G. Turk, technical director of the recently established Research, Product Development, and Standards Laboratory of the Paper Products Division.

Guy B. Clark will succeed Mr. Nelson as quality supervisor, it was announce by Fritz Masel, chief packaging and service engineer.

Mr. Nelson is active on several committees of the Technical Association of Pulp and Paper Industries and is currently heading a special study on "flat crush" of corrugated board. He has more than 27 years experience in the corrugated packaging operations at O-I, serving in Alton, Ill., and Charleston, W. Va., before moving to Toledo 10 years ago.

Knowlton

Frederick C. Keeney has joined Knowlton Brothers, Watertown, N. Y., manufacturers of specialized papers for industry, as assistant to the vice-president for production.

St. Regis

St. Regis Paper Co. has announced a $\$1^1/_4$ million modernization program at the Bucksport, Me., paper mill of its Printing Paper Division.

The program will include additions to the mill's wharf paper warehouse and trainshed, and improvements on the machine roll equipment of the mill's no. 1 paper machine. In addition, the building of a second-stage groundwood bleach plant is planned for completion sometime in November.

The new warehouse addition will be 125 by 213 ft. and will provide additional storage space for all types of publication and commercial printing papers made at Bucksport. Improvements on the no. 1 machine include a new reel and winder, new rewinder for the finishing room, new elevator for greater weight capacity, and a new Delavator for lowering rolls of paper from the machine room to the warehouse. These improvements will make it possible for the mill to increase the maximum diameter of its paper rolls from 40 to 50 in. in line with customer demand for paper rolls with larger diameters.

The planned second-stage bleach plant will make it possible for the present single-stage mill to produce pulp with greater brightness. The installation will be a "zinc-hydro" medium density type which will have a bleaching capacity of from 7 to 12% consistency.

The Verrier Construction Co., Portland, has the contract for and has already begun construction of the warehouse and trainshed. Most equipment for the improvement of paper production facilities will be supplied by the Improved Paper Machinery Co., Nashua, N. H.

Nekoosa

Construction will begin this week of a major remodeling project to house a new paper machine at the Nekoosa-Edwards Paper Co., producer of business papers.

The contract for construction work has been awarded to the Paul A. Laurence Co., Minneapolis. It involves installing a new roof over the present Nekoosa machine room approximately 15 ft higher and other structural changes to provide a greatly enlarged room to house an existing paper machine and the new no. 2 machine. A 25-ton, overhead, traveling crane will be installed for erection of the new ma-

chine and subsequent maintenance on no. 1 and 2 machines.

The new machine, largest of the company's 11 production units, will be built by Beloit Iron Works. It will have a 195-in. wire with 172-in. trim and a designed speed of 1500 f.p.m. Maximum production of the machine will be 170 tons per day.

By comparison, the company's largest existing machine is its no. 9 which was put into operation in December, 1952. This machine has a 180-in. wire, maximum trim of 166 in.

The new machine will employ a differential drive which permits better speed control than is possible with present belt-driving equipment. A new steam turbine has been purchased to drive the new machine.

Erection of the machine is scheduled to start in March, 1960, and will be completed in the fall. When in operation, it is expected to boost the company's over-all production of fine papers about 15 to 20%.

St. Lawrence

St. Lawrence Corp., Ltd., of Montreal, Que., has begun the first Canadian production of Clupak extensible paper, it is announced by G. E. Amerman, president of Clupak, Inc.

The new paper is being made on St. Lawrence's no. 4, 136-in. wide machine at its East Angus, Que., mill, and is being sold under the company's Bulldog trade name in conjunction with the Clupak trademark.

Capable of producing Clupak paper at the rate of approximately 140 tons a day, this new extensible unit will make available for the first time in Canada, duty-free Clupak paper for use in multiwall sacks, grocery bags, and converting kraft paper grades.

Watervliet

The Watervliet Paper Co., Div. of the Hammermill Paper Co., at Watervliet, Mich., has grown prosperous since its establishment in 1892 because its management policy has always been "production for quality." Constant progress has brought this mill to a 24-hr. daily production of more than 200,000 lb. of fine coated book and label paper.

Ten years ago, in the face of rising labor and equipment costs, the Watervliet papermakers decided to do something about cutting expenses through more efficient production. They sought the best way to eliminate old-fashioned processing methods that caused waste and slow-downs—such as wrinkles, cuts, prints, "snakes," slack edges, distortion, and shrinkage. They saw an advertisement in an industry paper for Mount Hope rolls, claiming to provide elimination of these costly papermaking problems, if the old-type brass rolls were replaced by the new Mount Hope Rolls.

Watervliet production men decided to try out Mount Hope rolls and an installation was made of a roll in the drying section of one of their fourdriniers to keep the paper under even tension and eliminate wrinkles in the drying paper. This roll did the job remarkably well and since then, on a consertive, step-by-step basis, Watervliet has become practically a 100% Mount Hope roll-equipped mill. In addition to the after-press operation in the drying section, other Mount Hope roll installations include those used on coating machine drum winders; at in-going and out-going calender nips, to keep sheet straight and step up output, and on their sheeters, where the rolls are used to give even tension and eliminate wrinkles, slack edges, baggy centers, and distortion.

Packaging Corp.

Formation of Packaging Corp. of America was announced today with the merger of American Box Board Co., Grand Rapids, Mich.; Central Fibre Products Co., Quincy, Ill.; and The Ohio Boxboard Co. of Rittman, Ohio.

Wayne Young, Packaging Corp. board chairman and former board chairman of Ohio Boxboard, said that the larger corporation will provide additional and improved facilities, greater integration, a wider product range, and a market area extending from New York to the Rocky Mountains.

He said increased product diversification and the expanded geographic area over which products are distributed will make the new company less vulnerable to economic decline in any one industry or locality.

"It will permit us to devote much more time, attention and money to research and development. All of this should add up to greater increased sales and better service to all of our customers," Mr. Young said.

At present, a large new kraft mill in which the new corporation holds a majority interest is under construction at Counce, Tenn. Also under construction is a new container plant at Lancaster, Pa. The new corporation begins operations with mills and converting plants at 42 locations in the East, Midwest, and West.

An initial nine-man board of directors approved by the stockholders include three from each of the merged companies.

The consolidation brings together facilities producing jute and kraft linerboard, boxboard, bleached kraft, corrugating medium, molded pulp products, egg packaging products, shipping containers and folding cartons, and other paperboard products.

"The new corporation unites the business leadership and outstanding productive abilities of three of the oldest and most successful independent companies in the packaging industry," according to Mr. Young. "They are essentially equal in size. All are strong in their respective regions. They are not essentially in competition with each other. Last year aggregate sales of the three firms approximated $120 million."

Twenty-five persons, including representatives from Mexico and Canada, are enrolled in the summer's second course on "Principles and Practice of Coated Paper Manufacture." The class runs from July 12 to August 1. Pictured are, left to right: First row—Erwin Heckman, Stein Hall & Co.; J. B. Dore, Ralston Purina Co.,; R. L. Janes, WMU faculty; P. S. Nathan, General Tire & Rubber Co.; L. R. Beeman, Watervliet Paper Co.; M. R. Barbe; Ruprecht Pirzer, Pulp & Paper Co.; and A. H. Nadelman, head of the WMU paper technology department.

Second row—D. Mai, T. F. Washburn Co.; R. L. Barker, American Cyanamid Co.; B. H. Landis, Clinton Corn Processing Co.; Jose Icaza; Hugh Baker, Olin Mathieson Chemical Corp.; Wilbur Evans, Champion Paper & Fibre Co.; D. J. Lynch, Armour & Co.; R. A. Diehm and John Fanselow, WMU faculty.

Third row—L. H. Reinke, Dow Chemical Co.; T. C. Morganstern, Minerals & Chemicals Corp.; C. F. Warner, P. H. Glatfelter Co.; W. C. Howard, Champion Paper & Fibre Co.; Gerrit Boas, Black-Clawson Co.; William Sharp, Union Sales Corp.

Fourth row—George Rasmussen, Consolidated Water Power & Paper Co.; J. P. Blaschka, Crown Zellerbach Corp.; D. D. Murison, E. B. Eddy Co.; J. C. Coulter, Anheuser-Busch Co.; J. R. Belche, Corn Products Sales Co., and L. W. McCallum, J. M. Huber Corp.

Twenty-six persons have completed the first of two 3-week courses in the "Principles and Practice of Coated Paper Manufacture," and offered by the department of paper technology, Western Michigan University. Pictured with the faculty, left to right, are: First row—M. Pavol, Minerals & Chemicals Corp.; Louis Dranikoff, Stein, Hall & Co.; Kenneth Damp, A. E. Staley Mfg. Co.; R. L. Janes, WMU faculty; Mack Williams, Mead Corp.; F. J. Farrell, Oil Mathieson Chemical Co.; John Harness, Hubinger Co.; A. H. Nadelman, head of the WMU paper technology department, and John Fanselow, WMU faculty.

Second row—Charles Mehlman, Clinton Corn Products Co.; William Ennis, Champion Paper & Fibre Co.; Gordon McCarrell, American Marietta Co.; R. A. Kraus, Corn Products Sales Co.; F. J. Slama, Corn Products Refining Co.

Third row—J. F. Triplett, Ohio Boxboard Co.; Forrest Peters, Simpson Paper Co.; John Grammel, Consolidated Water Power & Paper Co.; Frank Wilcox, Union Sale Corp.; Virgil Brecht, Central Fibre Products Co.

Fourth row—Kenneth Turner, Southern Clays, Inc.; Lee Strong, KVP Co.; Robert Gaiser, Crown Zellerbach Corp.

Fifth row—Gerald Schumann, Northwest Paper Co.; Joseph Latimer, Air Reduction Co.; R. A. Diehm, WMU faculty; Ralph DeMarco, Kimberly-Clark Corp.; Morris Cordova, St. Regis Paper Co.; Edward Norris, Champion Paper & Fibre Co.; H. T. Michael, Champion Paper & Fibre Co.; and Edward Persson, Black-Clawson Co.

For the present, administrative offices of the new corporation will remain in Grand Rapids, Mich., Quincy, Ill., and Rittman, Ohio, respective headquarters for each of the merging firms.

Stone Container

Stone Container Corp. has bought the business of Acme Carton Co., Chicago manufacturer of folding cartons and setup boxes.

Norman H. Stone, board chairman and president of Stone, who announced the purchase July 30, said that the operation will continue as Acme Carton Division of Stone Container Corp.

Lee Bernstein, who has been Acme's president, will be in charge of the division as general manager, Stone said, and no changes of personnel are planned. Terms of the purchase were not disclosed.

Acme Carton, founded 35 years ago, is located in a 90,000-sq.-ft. building at 5950–60 S. State St. It employs 200.

It becomes the third package-manufacturing division in Chicago for Stone Container, whose total operations include four container plants, five carton plants, and three paperboard mills, with 1958 sales of nearly $36,000,000.

In Chicago, Stone Container has a corrugated box plant at 4200 W. 42nd Pl., also company headquarters site, and a W. C. Ritchie Division at 8801 Baltimore Ave., which makes folding cartons, setup boxes, fiber cans and tubes and other packages.

"Besides expanding our sales volume, our purchase of Acme Carton further strengthens our position as a supplier of complete packaging service, backed up by our own paperboard mills as sources for raw materials," Stone said.

Fibreboard Paper Products

Fibreboard Paper Products Corp. will start construction immediately on an integrated shipping container plant in Phoenix.

"Fibreboard, serving the West with plants and sales offices throughout the 13 westernmost states, has long recognized the Southwest as a dynamic leader in both agricultural and industrial growth," William L. Keady, Fibreboard president said. "Our company has conducted a year-long study of the market in Arizona and New Mexico, and we have concluded that a shipping container plant should be built in Phoenix without delay."

Construction of the new plant will be completed by the end of the year, Mr. Keady said. Heart of the plant's operation will be an 85-in. corrugator, the first modern machine of its kind in Arizona. It will convert paperboard from Fibreboard's western mills into corrugated board for shipping containers.

Purchase of the Phoenix plant site is under active negotiation, and the location will be announced shortly, Mr. Keady said. The new plant will ultimately employ 90 people, the majority of whom will be recruited locally, he added.

The Phoenix container plant will be a facility of Fibreboard's Southern Packaging Division, which has maintained sales offices in Phoenix for more than 10 years. In Phoenix announcing the new plant construction was J. A. McDaniel, general manager of the Southern Packaging Division.

I.P.C.

The Fourth Industry Seminar, attended by 25 representatives of pulp and paper companies, was recently concluded at

Paper industry seminar June 14 to July 2, 1959. Seated: Dean R. P. Whitney, IPC; R. J. Shinners, Great Northern Paper Co.; A. A. Yankowski, Kimberly-Clark Corp.; R. L. Paret, East Texas P & P Co.; J. H. Galloway, Riegel Paper Corp.; H. A. Womack, Longview Fibre Co.; L. R. Giersch, Crown Zellerbach Corp.; R. Faessinger, Scott Paper Co.

Standing First Row: A. N. McLeod, seminar director; D. J. MacLaurin, associate seminar director; G. D. Jernegan, director of continuing education; C. H. Heidemann, Owens-Illinois Glass Co.; O. K. Sanders, Champion Paper & Fibre Co.; B. C. Miller, P. H. Glatfelter Co.; W. Henricks, The Mead Corp.; W. P. Newman, Jr., Continental Can Co., Inc.; B. McMahon, American Box Board Co.; W. L. Orr, St. Regis Paper Co.; P. G. Kort, Rhinelander Paper Co.; S. J. Ryczak, The Chesapeake Corp.

Standing Second Row: C. A. Langenbach, Crandon Paper Mills; J. W. Mollison, Inland Container Corp.; L. R. Vianey, Eaton-Dikeman Co.; H. A. Sutphen, Jr., West Virginia P & P Co.; F. G. Williams, Ontario Minnesota P & P Co. Ltd.; W. MacConnachie, Jr., Northwest Paper Co.; G. H. Marsh, Union Bag-Camp Corp.; H. B. Brown, Central Fibre Products Co.; J. W. Mengel, Eastman Kodak Co.

The Institute of Paper Chemistry. The three-week program, directed by A. Neil McLeod, institute economist, was designed to promote an increased awareness of the contributions to be made by science and technology to this industry and to broaden the perspectives of the men in attendance. Also, consideration was given to the resources of the industry and to the historical impact of geographic, economic, political, and social factors which have brought the industry to its present state of development.

In addition to the staff and student presentations and activities, feature speakers were: John Wollwage, manager of foreign operations, Kimberly-Clark Corp., Neenah, Wis.; Malcolm K. Whyte, Whyte, Hirshboeck, Minihan, Harding and Harland, Milwaukee, Wis.; and Malcolm Dole, Department of Chemistry, Northwestern University, Evanston, Ill. Their subjects were: "Foreign Operations in the Paper Industry," "The Impact of Modern Legal Developments on the Making of Paper," and "The Outlook for Industrial Application of Atomic Energy," respectively.

Participants resided in two Lawrence College fraternity houses and attended sessions in the Institute classrooms.

Ten professors of chemistry from colleges throughout the country did research at The Institute of Paper Chemistry this summer on stipends from the National Science Foundation. The Research Participation Program for Teacher Training, directed by Dean Roy P. Whitney, is designed to stimulate research activities in the small colleges. Specifically the program provides opportunity for chemistry teachers to make library studies, plan research programs and carry out actual research in selected fields under the guidance of senior members of the Institute staff. It permits the participants to become familiar with and use experimental equipment and instruments beyond those normally available on the small college campus.

The program, which began on July 7 continued through August 29, including weekly seminars and staff consultation.

Wisconsin

The University of Wisconsin Industrial Packaging Institute announces that its Second Annual Industrial Packaging Institute will take place on Thursday and Friday, Oct. 29 and 30, 1959.

The initial packaging institute took place in September of last year and the interest shown by industry was most gratifying. Over a hundred people were in attendance from manufacturing plants not only in the Mid-West but ranging out to the East Coast and Southern states. The program at that time was more or less experimental, but sufficed to point the way for future packaging institutes to be presented by the University of Wisconsin.

The cost reduction aspects of packaging were touched on lightly during last year's program. This year more emphasis will be applied in this direction as well as exploring to a greater extent the relative obligations of top management and the packaging engineer toward a more efficient packaging program. The University of Wisconsin hopes to present not only general discussions, but smaller group clinics on specific aspects of industrial packaging. The range of products to be considered in this area will cover the packaging of liquids and flowable solids through problems of merchandising and prepackaging of hard goods. The increasing significance and impact of plastic materials in the packaging field will also receive attention at this time.

The speakers and group leaders will be selected with great care. It is intended that these men will present to industry packaging thoughts and ideas revolving around the more important aspects of industrial packaging as they exist today.

The packaging institute this year as last, will be held in the new Wisconsin center building on the main campus of the University of Wisconsin. This beautiful new building was created specifically for adult education programs and is admirably suited for an institute of this nature. The Institute of Industrial Packaging represents recognition on the part of the University of Wisconsin on the manifold problems confronting industry today.

Further information may be obtained by writing Richard A. McCormick, Institute Coordinator, 4016 Stadium, the University of Wisconsin, Madison 6, Wis.

The University of Wisconsin Industrial Adhesives Institute in cooperation with the Glues and Gluing Division of Forest Products Research Society and the Forest Products Laboratory announces that its institute "Industrial Adhesives Applications" will take place on Thursday and Friday, Oct. 1 and 2, 1959. Companies using or considering using adhesives in the bonding of wood, metal, or plastic will find in this institute solutions to problems encountered in design and fabrication with adhesives, trouble shooting adhesive bonds, and future uses for adhesives to improve product quality or reduce product costs.

Outstanding speakers from industry and the forest products laboratory will present the most progressive and important views on industrial adhesives applications. The meeting will be held in the new Wisconsin center building on the main campus of the University of Wisconsin.

Further information may be obtained by writing Richard A. McCormick, Institute Coordinator, 4016 Stadium, the University of Wisconsin, Madison 6, Wis.

Research participation program for teacher training. Front row: H. F. Lewis, IPC; R. G. Werth, Concordia College, Moorhead, Minn.; A. J. Bosch, Central College, Pella, Ia.; Gerrit Levey, Berea College, Berea, Ky.; E. W. Lewis, Texas Western College, El Paso, Texas; D. A. Scott, Drew University, Madison, N. J.; Herman De Haas, Westminster College, New Wilmington, Pa.; K. L. Lockwood, Western Maryland College, Westminster, Md.; S. L. Burson, Pfeiffer College, Misenheimer, N. C.; J. L. Dalton, St. Ambrose College, Davenport, Ia.; H. E. Kirkby, Shimer College, Mount Carroll, Ill.

Back row: R. P. Whitney, IPC; S. F. Darlig, Lawrence College; D. G. Williams, IPC; E. O. Dillingham, IPC; J. W. Green, IPC; J. W. Swanson, IPC; L. E. Wise, IPC; I. A. Pearl, IPC; Kyle Ward, Jr., IPC; M. A. Buchanan, IPC; G. D. Jernegan, IPC.

Perkins-Goodwin

Louis Calder, Jr., president of Perkins-Goodwin Co., international pulp and paper marketing-management organization, has announced the election of Francis X. Donlan as a vice-president.

Mr. Calder also announced that Stanford G. Blankinship, who observed his 65th birthday on July 23, is retiring from active service with the company. At the request of the company he will continue as a vice-president and director, serving in an advisory capacity to the pulp department. Well known throughout the industry, Stan Blankinship was associated for 25 years with the Brown Co. in Berlin, N. H., and from 1929 to 1941 was manager of the Brown Co.'s pulp mills. He is a past president of the American Pulp and Paper Mill Superintendents Association and the U. S. Wood Pulp Importers Association. He joined Perkins-Goodwin Co. in 1945 as a

vice-president, and since that time has headed the pulp sales department.

Chromalox

A new catalog illustrates and describes the complete line of Chromalox far-infrared heaters and controls. These heaters, using the far-infrared wavelength, are used for the drying, curing, and baking of plastic, textiles, paper, and inks, ceramic products and for metal preheating work, glass, and many other uses. For further information write, Edwin L. Wiegand Co., 7500 Thomas Blvd., Pittsburgh 8, Pa.

Wallerstein

The Wallerstein Co. celebrated the opening of its new administration building at Mariners Harbor, Staten Island, on Saturday afternoon, May 23, with a Family Day Open House for all employees and their families.

The administration building, opened on May 4th, houses the executive, sales, and general offices of the Wallerstein Co. For the first time in 40 years the offices, manufacturing facilities, and laboratories are located together at the plant site at Wallerstein Square in Mariners Harbor. This grouping of the executive, technical, and production departments will provide Wallerstein with an opportunity for even greater service to its customers.

Over 1600 guests—employees and their families, their children, grandchildren, relatives, and friends—streamed through the attractive entrance of the ultramodern building.

The recent construction and opening of the new administration building is another step forward in Wallerstein's development program for the future. From its beginning in 1902 as a small analytical laboratory, the Wallerstein Co. has grown to its present leading position as an authority on the industrial application of enzymes. Its enzymatic products are used in the brewing, leather tanning, textile, baking, pharmaceutical, meat packing, paper, and dry cleaning industries.

Pfaudler Permutit

A new 16-page "Fluidics" Buyers Guide of interest to all companies who handle or process liquids or gases is now available from Pfaudler Permutit, Inc., Rochester, N. Y.

The new bulletin explains fluidics—the science of fluid processes—as a new Pfaudler Permutit program providing a modern, imaginative approach plus the specialized materials and equipment for handling and processing more profitably the liquid and gases used by industry. Pertinent applications range from reactions to storage, from water treatment to waste treatment, from design and development to project engineering.

Sprout, Waldron

The availability of a new bulletin on 30 and 42 in. pressurized refiners has been announced by Sprout, Waldron & Co., Inc. Bulletin 212 covers the design, construction, and operation of the Sprout-Waldron balanced thrust pressurized refiner with parallel feed arrangement. Copies are available on request.

S & S Corrugated Paper Machinery

S & S Corrugated Paper Machinery Co., Inc., has appointed Nuttall Engineering Pty., Ltd., exclusive agents for sales, installation, and maintenance of S & S machinery in Australia and New Zealand, according to an announcement by Mitchel Flaum, president of the Brooklyn, N. Y., firm.

Mr. Flaum added that Nuttall also will manufacture, under license, certain machines of S & S design, under the direction of S & S resident engineers. As is the practice with all S & S machines manufactured overseas, the Nuttall-built machines will be identical to S & S machines manufactured in Brooklyn.

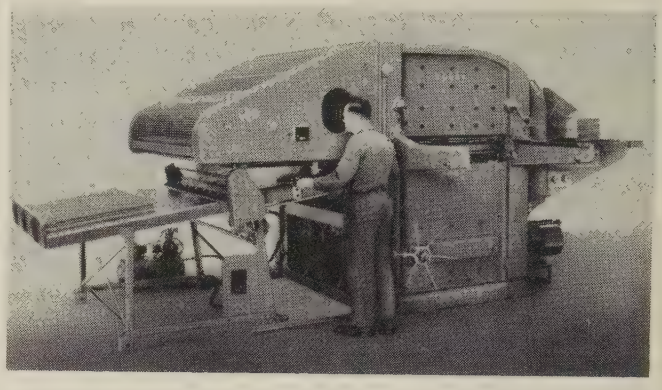
The announcement followed a recent tour and study of S & S manufacturing methods by William Nuttall, director of the 35-year-old Australian firm.

The Nuttall office and plant are located at 9 Primrose Ave., Rosebery, Sydney, N.S.W.

Thomson-National Press

A new 42 by 62 corrugated and solid fiber cutting and creasing press, the Bobst SPO 1575 Autoplaten, is now available from Thomson-National Press Co., Inc., Franklin, Mass.

The SPO 1575 42 by 62 offers press speeds up to 4500 sheets per hour and is a special new model without stripping stations.



New Bobst cutting and creasing press

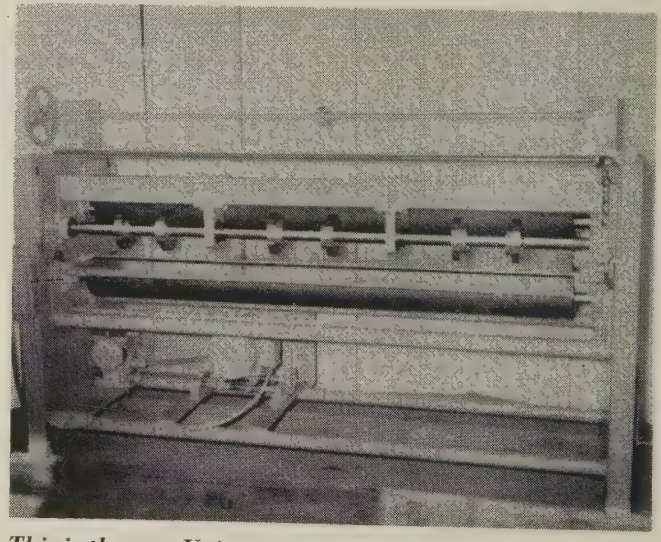
This press features a pivot-in-place cutting chase which can be pulled out horizontally and turned over for easy access to makeready without removing the chase from the press.

Cutting, creasing, and stripping makeready can be performed away from the press for one job while another job is running. A new Bobst makeready table for stipping preparation away from the press is now available. Cutting and stripping makeready can be saved and replaced in the press in perfect register for reruns.

The SPO 1575 handles corrugated stock up to $^3/_8$ in. and fiberboard in all normal thicknesses. Either a 0.937 or $1^1/_8$ in. cutting rule can be used with no crushing of the stock. Finished sheets are counted in piles and fed into the new Bobst roll-off delivery table.

Union Tool

A strip roller coater that meets the high production requirements of the carton, paper, and wood industries has been



This is the new Union strip coater designed to apply adhesive in given strips and patterns to many different types of materials. It is built by The Union Tool Corp., Warsaw, Indiana

developed by Union Tool Corp. The Union strip coater permits adhesive application in given strips and patterns to many different types of materials. The above unit is being used to apply glue for cleats to corrugated cardboard.

Strip rolls are adjustable for different patterns of adhesive and can be interchanged for wide or narrow glue strips as desired. Openings in the upper coating roll assemblies are adjustable to 2 in. through the gear-elevating mechanism with single handwheel control. A variable speed drive is also available with any 3 :1 ratio.

Coating assembly consists of three rolls: a doctor roll, a transfer roll, and a strip roll: The adhesive is carried in the crotch between doctor and transfer rolls. Thick or thin coatings can be applied by a simple adjustment of the openings between these two rolls. Further control of the adhesive thickness can be obtained by running the strip rolls in and out toward the transfer rolls.

The Union strip coater is available with rolls from 12 to 108 in. in length. Unit illustrated is a top strip coating machine; bottom type units are also available.

Complete details are available from Union Tool Corp., Warsaw, Ind.

Appleton Machine

Appleton Machine Co. announces formation of a European subsidiary named Appleton International S.P.A. The decision to open foreign offices, located in Milan, Paris, London, and Frankfort, came as a result of increased demand for papermaking machinery in these countries and customer service on existing Appleton equipment in Europen mills.

Milos Krofta, internationally known engineer and designer of pulp and paper mill machinery, has been appointed to supervise the overseas operation.

Appleton International will conduct all sales, service, and manufacture of its parent firm's supercalenders, machine calendars, paper mill and converting winders, slitters, and stock preparation equipment. It will also represent the Gilbert and Nash Co. covering the sale of air, mechanical, felt, and wire paper machine guides.

J. M. MacDonald, Jr., Appleton Machine vice-president, will direct liaison between the two companies.

"Our name is well known to the European papermaker," said Mr. MacDonald. "Engineers from foreign countries have toured American pulp and paper mills and have seen our equipment in 95% of them. In addition, considerable Appleton equipment has been in service for a long time in many paper mills overseas," he added.

Headquarters for the European subsidiary will be: Appleton International S.P.A., Via Fontana 11, Milano, Italy.

Bauer

The Bauer drainer conveyor for conveying and dewatering wood chips, defibered pumpable stock, or similar materials is described in Bulletin P-29, now available from The Bauer Bros. Co., 1715 Sheridan Ave., Springfield, Ohio.

A four-page bulletin describing the newly introduced no. 444-24" pump-through refiner is now available from the Bauer Bros Co., 1715 Sheridan Ave., Springfield, Ohio.

Thwing-Albert

New information Bulletin 373-ST describes the Fotosize Liquid Penetration Tester—the culmination of 10 decades of developing and building penetration testers by the Hercules Powder Co.—for determining the sizing of paper.

A copy of Bulletin 373-ST may be obtained from the Thwing-Albert Instrument Co. at Penn St. & Pulaski Ave., Philadelphia 44, Pa.

The capacity of the Elmendorf tearing tester has been increased to 6400 grams in the textile model of the Elmendorf tearing tester. The textile model has a capacity four times that of the standard model elmendorf tearing tester.

The textile model is a three-range instrument with a basic range of 1600 grams. It can be used as a 3200-gram instrument by adding one augmenting weight to the pendulum. By adding two augmenting weights it can be used as a 6400-gram instrument. Because each range is calibrated separately the augmenting weight and textile weight can be slipped on or off without affecting the calibration of the instrument.

Developed originally for the paper industry, the Elmendorf tearing tester is used in other industries where the internal tearing resistance of a sheet material, such as textiles, plastics, metal foils, etc., is important.

The textile model Elmendorf tearing tester is made by the Thwing-Albert Instrument Co. at Penn St. & Pulaski Ave., Philadelphia 44, Pa.

Bulletin 371-ST on standard and water-jacketed Penescope has just been completed. The Penescope is probably the simplest and most inexpensive instrument for testing the resistance of paper, paperboard, plastic films and other materials to penetration by water, inks, blood, and solutions of electrolytes and other liquids. Bulletin 371-ST is available from the Thwing-Albert Instrument Co. at Penn St. & Pulaski Ave., Philadelphia 44, Pa.

Koppers

A new latex for pigmented paper coatings which has greater adhesive strength and less foam has been announced by the Plastics Division of Koppers Co., Inc. Known as Dylex Latex KCD-154, this new product is available in commercial quantities although still in the developmental stage.

Improvements over conventional styrene-butadiene latices have been obtained by carefully adjusting the emulsifier content and by fortifying the polymer during polymerization.

The new latex is reported to have as much as 50% increased film strength, which results in better pigment binding and increased pick resistance of the coated sheet.

Experimental work on Koppers' large pilot coater located in the company's Kobuta development laboratory indicates that use of this new latex increased pick resistance at all adhesive levels checked, whether it was in combination with starch or casein.

In addition, foaming tendencies are reported to be considerably decreased. There is considerably less foam formed initially during agitation with other coating ingredients, and this minimal foam dissipates at a much more rapid rate than in conventional latex.

Compatibility with various clays and pigments as well as with starch and casein is reported to be excellent.

A new bulletin with further detailed information concerning this new latex has just been issued. Copies may be obtained by writing to the Plastics Division, Koppers Co., Inc., 801 Koppers Building, Pittsburgh 19, Pa., and asking for Technical Bulletin C-9-28%.

Diamond Alkali

Appointment of Robert B. Turk to the staff of Diamond Alkali Co.'s pulp and paper technical service laboratory was announced by Claud B. Christiansen, supervisor of pulp and paper technical service.

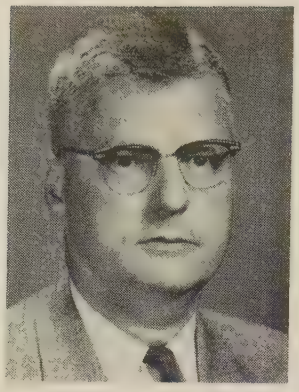
At his new post Mr. Turk will supervise laboratory projects and technical service assignments for Diamond customers.

Cyanamid

A new liquid dye for paper, Green MX Liquid, has been added to American Cyanamid Co.'s line of liquid paper dyes.

The new product is specifically designed to eliminate the dusting and handling problems that result from the use of some powder types. Since dust from dye particles in the air can stain the body, paper mill technicians who weigh or measure dye often under hot, humid conditions will appreciate its dust-free nature.

S. G. Briscoe, The Pusey & Jones Corp.



J. P. Casey, Union Starch & Refining Co., Inc.

PUSEY & JONES

The Pusey and Jones Corp. of Wilmington, Del., manufacturers of paper machinery and other fabricated steel products, announced the appointment of Sidney G. Briscoe as vice-president-sales.

UNION STARCH

The appointment of James P. Casey to the position of director of starch sales of the Union Starch and Refining Co. Inc., and Union Sales Corp. was announced by T. R. Reed, executive vice-president.

Mr. Casey will assume the responsibility for the sale of the company's line of starch products to consuming industries throughout the United States. The company markets a variety of starches that are used by the textile, pulp and paper, food, and confectionery industries, among others.

FITCHBURG SCREEN PLATE

The Fitchburg Screen Plate Co., Inc., Fitchburg, Mass., recently purchased the Wickwire Spencer Perforating Div. of Colorado Fuel and Iron Corp. The new company bears the name National Perforating Corp. and is located at Parker St., Clinton, Mass. Sales and administrative offices are located at 309 South St., Fitchburg, Mass., offices of the parent company.

National Perforating manufactures a wide variety of perforated metals, plastics, paperboard, and other materials for many industries, including chemicals, mining, textiles, furniture, electronics, aircraft, and shipbuilding, as well as decorative panels for the construction industry.

The new perforating plant provides complete facilities for the manufacture of all types of perforated centrifugal and pressure screen plates for the pulp and paper industry in a wide variety of metals, including stainless steel, inconel, monel, copper, and aluminum bronze. Sales to the pulp and paper industry will be handled through Morey Paper Mill Supply Co., 309 South St., Fitchburg, Mass., which is an affiliated company under the same management.

BOLTON

The resignation of Haigh M. Reiniger from John W. Bolton & Sons, Inc., and its Emerson Manufacturing Co. Div. has been announced. Mr. Reiniger was vice-president in charge of sales since joining the company in 1954.

F. Richard Convey, Northeastern region manager has been appointed general sales manager.

John W. Bolton, III, has been promoted to director of marketing services. In his new position Mr. Bolton will handle all internal sales administration for the company's line of stock preparation machinery and equipment.

MALLORY-SHARON

A major titanium product development program for the pulp and paper industry has been inaugurated by Mallory-Sharon Metals Corp., Niles, Ohio.

The program will seek to develop and evaluate titanium and titanium alloy products for pulp and paper processors.

According to Graham B. Brown, vice-president of Mallory-Sharon, the development program is being launched as a result of the many applications which have already been found for titanium in this industry.

Titanium or its alloys is now being used in pulp and paper chlorine dioxide reactors, scraper arms, pipe strapping, and other components where long service life in extremely corrosive situations is a necessity. Titanium parts in such applications are showing no corrosive attack after as much as a three-year service, compared to other materials which survive as little as two weeks.

The product development program for the pulp and paper industry will represent a joint effort on the part of Mallory-Sharon's product development and engineering departments.

BURGESS-MANNING

Water sealed vacuum pumps usually require independent water separators and silencers when the discharge noise is excessive or objectionable. The Burgess-Manning water separator snubber performs both functions efficiently.

During the installation of a vacuum pump at a Wisconsin paper plant, a series of tests were made to determine the efficiency of a water separator snubber in the reduction of discharge noise of the pump. Over all sound level readings and half octave band analyses were taken at a point adjacent to the discharge outlet both before and after the snubber installation.

Tests were run with the vacuum pump outlet pipe open to atmosphere with the vacuum pump operating at 257 r.p.m. with 28 g.p.m. of seal water. The discharge velocity was 5100 f.p.m. and the fundamental frequency was $85\frac{1}{2}$ c.p.s. The over-all noise level was considered to be higher than would be recommended in a satisfactory working area.

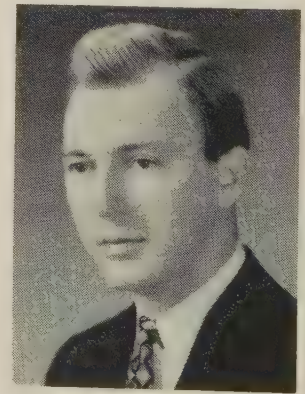
The Burgess-Manning Water Separator Snubber was then installed and tests showed an attenuation of 18 db. of the over-all noise level.

The fundamental frequency was reduced 12 db. and noise previously attributed to a first harmonic was entirely eliminated.

Water separation at 28 g.p.m. was entirely satisfactory and the installation demonstrated the efficiency of the water separator snubber in both its water separation and silencing functions. Additional information about the dual purpose snubber may be obtained from Burgess-Manning Co., Industrial Silencer Div., 9207 Sovereign Row, Dallas 35, Tex.

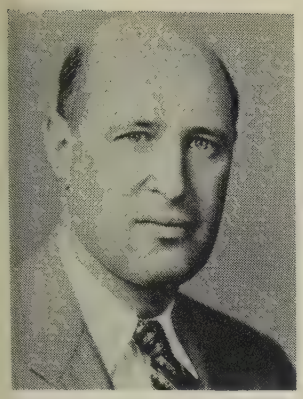
HUYCK

Huyck Felt Co., Rensselaer, N. Y., and Aliceville, Ala., has announced the appointment of Frank S. Winship as felt sales engineer and William L. Korn as field service engineer.

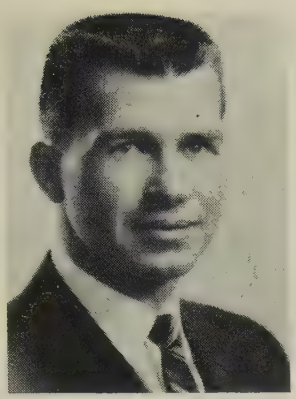


F. S. Winship, Huyck Felt Co.

W. L. Korn, Huyck Felt Co.



R. J. Jacobs, John Waldron Corp.



B. F. Howell, Arnold, Hoffman & Co., Inc.

MIDLAND-ROSS

Midland-Ross Corp. has announced the appointment, effective August 1, of Robert J. Jacobs as general manager of its subsidiary, the John Waldron Corp. of New Brunswick, N. J., and of its Hartig Extruder Division of Mountainside, N. J.

ARNOLD, HOFFMAN

Appointment of Benny F. Howell to serve the paper trade in the Southeastern market has been announced by the sales department of Arnold, Hoffman & Co., Inc., manufacturers of organic chemicals. The appointment, according to management, is keyed to the company's expanding sales of defoamers and other specialties developed especially for pulp and papermaking.

KELCO

Richard Markham has been appointed technical sales representative for the Paper Division of the Kelco Co., manufacturers of algin products with sales offices in New York, Chicago, and Los Angeles, and plant in San Diego, Calif.

G.E.

A new controlled atmosphere facility at General Electric's power tube plant here is improving the reliability and life of metal electron tubes used in paper mill drives.

This is believed to be the first time atmosphere control has been used in the production of metal electron tubes, although it has long been a requirement in the manufacture of television camera tubes and many types of receiving and transmitting tubes.

Three lines of metal tubes are being made in the new facility: ignitrons, thyatrons, and phanotrons. These versatile tubes are also used in applications such as steel mill drives, electric locomotive drives, welding control panels, radar modulators and motor control centers.

According to D. C. Scott, manager of the Schenectady tube plant, the new facility was designed to increase tube reliability by preventing undetected contamination from being sealed into tubes.

General Electric's first showing of an industrial mass flowmeter which measures liquid and gas flow directly in pounds has been scheduled for the Instrument Society of America Conference and Exhibit, September 21–25 in the International Amphitheater, Chicago.

The device will headline an exhibit also to feature a new line of electronic process recorders and recording controllers, plus two other products being introduced by the company's Instrument Department.

These are a low-cost, portable leak standard for calibrating halogen-sensitive leak detectors, and an advance-design mass spectrometer leak detector.

The flowmeter represents the climax of a five-year research program aimed at developing a simple, low maintenance instrument for measuring the weight of flowing fluids with high accuracy over wide ranges of flow rate, pressure, temperature, and density.

A high speed, explosionproof direct-current motor utilizing a new ventilation system has been developed by General Electric's Direct Current Motor and Generator Dept. in Erie, Pa.

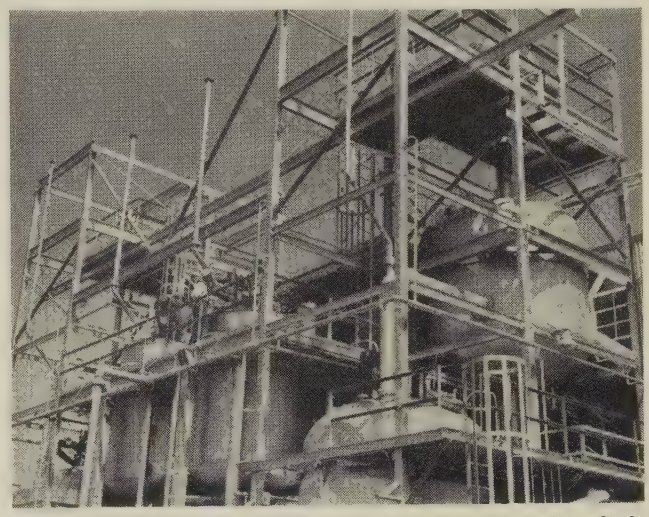
The new motor, with ratings from 40 to 150 hp. and speeds up to 6000 r.p.m. is another first in the company's line of Kinamatic direct-current motors.

Featuring a new water-to-air heat exchanger, which is more efficient than the conventional air-to-air heat exchanger, the motor will have a reduced over-all resulting in space saving to the user.

GENERAL TIRE

The General Tire & Rubber Co. has completed a $350,000, 50% expansion of its facilities for the production of paint, paper, and textile latices at Mogadore, Ohio.

General Tire, third largest producer in this field and second in number of customers, has also augmented its tank car fleet to take care of the additional production.



Facilities expansion of The General Tire & Rubber Co.'s Mogadore, Ohio, chemical plant is shown in this general view of the new latex reactor area

The product, a styrene-butadiene latex called Gen-Flo, is used in rubber-base latex paints, in new, improved coated papers as a binder and reinforcing agent, and in the textile industry in rug backing, textile printing inks and in the treatment of upholstery materials.

The additional facilities, which include a complete new glass-lined reactor system, also provide for production of styrene-acrylic latices, called Acri-Flo, for newly developed exterior latex paints.

DU PONT

Plans for the Du Pont Co.'s first major plant for production of polyethylene film for packaging were announced in Richmond, Va., August 10. Installation of production equipment will start immediately, and the plant is to be ready to begin production early in 1960.

Du Pont's polyethylene film pilot plant at the Sabine River Works, Orange, Tex., is now turning out packaging film which combines high impact strength with excellent appearance and printability, but this film will be available only in limited quantities until start-up of the new facilities at Richmond.

The company is now producing polyethylene film for industrial and agricultural applications in commercial quantities at its Yerkes film plant in Buffalo, N. Y.

To be operated as a unit of the present Spruance cellophane plant, the new production facilities will be located for the

most part in existing buildings formerly used for rayon production. A 12,000 sq. ft. addition to these buildings will be constructed for production equipment. New facilities will be provided for testing laboratories of the Spruance textile fibers plant, which are now situated in the buildings to be used for polyethylene film manufacturing.

STEIN, HALL

Roy C. Charron (Col., U. S. Army, Ret.) has joined Stein, Hall & Co., Inc., as adhesive specialist, Technical Division. This assignment will make his 38 years of adhesive research and development experience available to the industry on a nationwide basis through the company's technical sales service organization.

B & W

Robert E. Halloran has been named Pacific Coast sales manager of The Babcock & Wilcox Co.'s Tubular Products Division, it has been announced by Leon B. Wohlgemuth, general sales manager of the division. The appointment became effective August 1.

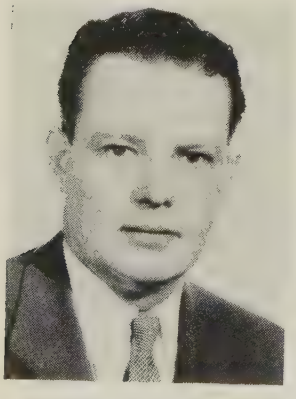
Mr. Halloran, who will make his headquarters at the Los Angeles district sales office, succeeds H. F. Lefferty, who is retiring after more than 40 years of continuous service with Babcock & Wilcox.

Fabricators who are confronted with the problem of joining tubing of similar or dissimilar types of steel by welding will benefit by the information contained in a new data card released by the Tubular Products Division of The Babcock & Wilcox Co. The data card summarizes the various recommendations for arc welding. The data card, identified as TDC-155, is available without charge from the general sales offices of the division at Beaver Falls, Pa.

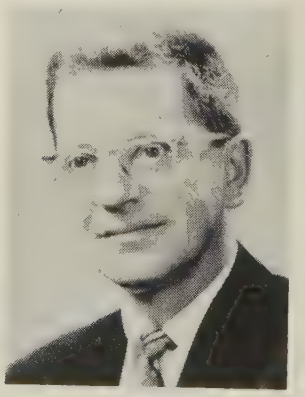
H. F. Lefferty, The Babcock & Wilcox Co.

Welding engineers and others associated with the fabrication and installation of process piping and tubing will benefit by the information contained in a folder released by the Tubular Products Division of The Babcock & Wilcox Co. Known as TDC-162, the publication provides information on the welding characteristics of both austenitic and ferritic types of stainless steel. Copies of the folder may be obtained without charge from the sales offices of the company at Beaver Falls, Pa.

Thomas W. Wright has been named assistant manager of the New York district sales office of The Babcock & Wilcox Co.'s Tubular Products Division.



R. E. Halloran, The Babcock & Wilcox Co.

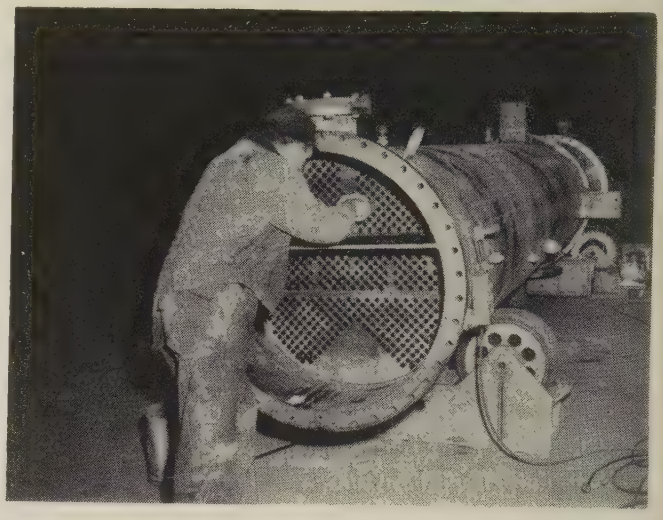


T. W. Wright, The Babcock & Wilcox Co.

C. B. & I.

Reclaiming valuable heat from spent sulphate black liquor vapors is a money-saving proposition with a 2600 sq. ft. o. d. vapor condenser now being built by the Chicago Bridge & Iron Co. for a pulp and paper mill in Brazil.

The vapor condenser is designed to condense distillation products out of a hot vapor line from a multiple-effect evapo-



rator. At the same time, the heat of these vaporous products is transferred at the rate of over 19 million B.t.u.'s per hour to incoming fresh water in the condenser tubes, preheating it to 25 to 30°.

By preheating the incoming water, steam needs for water heating are lowered, evening out the operating load on the plant's boilers and cutting fuel consumption. The recovery of 19 million B.t.u.'s per hour represents a saving of about $10 per hour or approximately $40,000 savings per year.

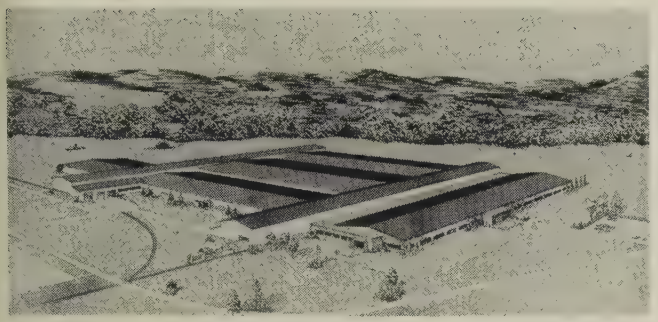
Here's how it works. Vapors and entrained noncondensables from the last effect evaporator are piped at the rate of 50,000 c.f.m. to the shell of the vapor condenser. Entering the shell at 130°F., these products contact the condenser's tubes and are subject to the condensing action of 1450 g.p.m. of cooling water passing through the tubes in a 3-pass circuit. The condensed evaporation products leave the shell at 130°F., having transferred 19 million B.t.u.'s per hour to the cooling water.

The presence of noncondensable sulphides in the vapor from the evaporator prevents the direct injection method of preheating the incoming water. Thus, a tubular exchanger is required. The hot vapors are piped into the shell of the vapor condenser while the incoming water is in the tubes. As the water passes through the tubes, it extracts heat from the vapors.

Because these noncondensables are corrosive to ordinary tubing materials, the fabricator selected type 304 stainless steel tubing which was supplied by the Alloy Tube Division of The Carpenter Steel Co. Several other materials were tested first. Carbon steel suffered rapid build-up of iron sulphide scale on the o. d. of the tubes and oxidation on the I. D. Chrome plated carbon steel and copper alloy tubes likewise failed to give satisfactory evidence of long service life. Use of type 304 stainless insures minimum down time and maintenance because it resists scale formation and oxidation.

JONES

The new plant of the E. D. Jones Corp. in process of being built is located along the Boston & Albany division of the NYCRR four miles east of the center of Pittsfield. This new facility on 82 acres of land astride the Pittsfield-Dalton line is due for completion about December of this year. The office and all manufacturing operations located in various parts of Pittsfield, principally in several multistory buildings in the



Sketch of the new E. D. Jones Corp. being built near Pittsfield, Mass.

center of the city, are to be moved to this single-floor operation. All buildings are interconnected and arranged so that there is a natural flow of materials from rough state through the processing bays to the assembly shop.

Incoming materials received either by truck or rail are stored to feed directly into multiple fabricating or machining bays each 50 ft. wide by 300 ft. long. Completed parts are stored adjacent to the assembly shop thus reducing in-plant movement to a minimum. Outgoing products are to be loaded at undercover rail or truck docks at the south end of the assembly shop. Wood processing, which is still a substantial part of the company's products, is to be handled in a building connected to the assembly area but away from other manufacturing because of fire hazard.

Total area under roof is about 195,000 sq. ft. including office. All construction is steel frame with clear spaces from 50 to 100 ft. and heights from 16 to 40 ft. Stores and all manufacturing areas are served with cranes varying in capacity from 3 to 25 tons. All roof areas are insulated with glass wool.

Basic planning for the buildings has been a cooperative venture by E. D. Jones Corp., various vendors and Beloit Iron Works. All phases of construction are proceeding with on-the-scene supervision of William Sorenson and his staff.

Miles Chemical

A major corporate realignment encompassing the formation of a new company which will be prominent in the paper field has just been announced by Miles Laboratories, Elkhart, Ind.

The new organization has been named the Miles Chemical Co. and is one of four main divisions of the parent corporation.

It consolidates into one the Sumner Chemical Co., Zeeland, Mich., makers of fine organic chemicals; Takamine Laboratory Division, Clifton, N. J., manufacturers of industrial enzymes; and citric acid production, Elkhart, Ind.

In the past, Takamine has been active in the paper industry through the sale of enzymes for modifications of starch to impart desired sizing and coating quality. Sumner is also active here with polymeric dialdehyde for increasing wet strength. Present plans call for increased activity in this area.

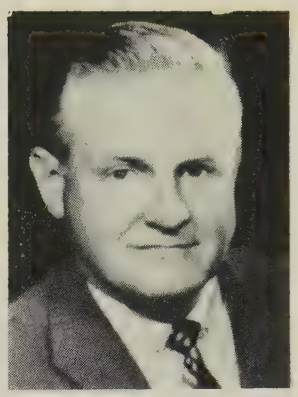
Howard F. Roderick has been named president of the new company. Mr. Roderick joined Miles Laboratories in January 1959 as vice-president and most recently was named to the board of directors.

Administrative and marketing headquarters under the new arrangement will be in Elkhart, Ind.

In making the announcement about the realignment, Miles Laboratories President Walter R. Beardsley said, "The move is being taken at this time to insure optimum efficiency, both internally and externally. The anticipated growth of this division, coupled with the complexity and variety of its products, is bringing about the need for a high degree of autonomy.

A new chemical product which could have far reaching application in the paper industry will soon be available in semicommercial quantities from Miles Chemical Co., Elkhart, Ind.

The product, dialdehyde starch, being marketed under the name Sumstar, is currently being evaluated for a variety of industrial applications with particular emphasis on the role it might play in the paper industry.



H. F. Roderick, Miles Chemical Co.

Originally developed by the United States Department of Agriculture's Northern Research and Development Division, the process has been refined and adapted for manufacture by Miles through an extensive independent research program.

According to John Mirza, director of chemical research and development, Miles Chemical Co., research to date has indicated that its use in the paper industry could be significant.

Dr. Mirza said that investigations have revealed that use of Sumstar can increase paper wet strength substantially. At the same time such characteristics as oil and grease resistance, tensile strength, elongation, stiffness and porosity may be altered desirably.

In addition to uses based upon its aldehyde function, Sumstar may be used either alone or with other materials as a sizing agent. A typical method of sizing paper to obtain wet strength is to pass the paper through an aqueous solution of Sumstar of about 5% concentration, remove excess solution (which may be refortified and reused) between rolls, and dry. If greater water resistance is desired, the paper may be subjected to a mild heat cure, Dr. Mirza said.

Another potential application for Sumstar in the paper industry may be as a binder for paper coatings in combination with casein, starch, and other natural or synthetic materials.

Certain derivatives, such as the urea, melamine, bisulphite, and acrylamide adducts are expected to be useful additives for the paper industry, according to Dr. Mirza.

Plans for bringing the product from its current pilot plant operations to semicommercial production were outlined by L. D. Williams, sales manager, Sumner products.

He said that a new semicommercial plant with a capacity of several hundred thousand pounds a year will be on stream by mid-October. Provisions are also being made for rapid expansions as needed.

Williams said that interest in the new product for the paper industry is encouraging. He said that working samples have gone out to the field where the performance of the product is being valuated.

At this time, pilot samples up to 5 lb. are available at no charge. Larger pilot quantities are available at $1.50 per pound.

When the new plant is on stream, prices will be reduced to $1.25 per pound for 100-lb. drums and $1.00 per pound for 1000 lb. and up.

Williams stated that Miles Chemical long-range plans for Sumstar point to a multimillion pound plant producing material to sell for 30 to 40 cents a pound with even further price reductions possible.

Three types are being offered: Sumstar-S (over 90% oxidized), Sumstar-R (75 to 80% oxidized), and Sumstar-J (50% oxidized). Special tailoring to fit specific applications can be arranged.

Sumstar is a fine powder, almost colorless, nonvolatile, odorless, and of extremely low acute toxicity.

Borden

Appointment of Maurice S. Letourneau as New York district sales manager for the Polyco-Monomer Department of the Borden Chemical Co. has been announced by W. G. West, sales manager.

OBITUARIES

William Thomas Kirby

William T. Kirby, Chemist for Crane & Co. Inc., Dalton, Mass., died as the result of an accident on July 22, 1959, at Dalton.

Mr. Kirby was born in Pittsfield, Mass. on Nov. 13, 1912, and attended Clark University. He joined the Crane organization in 1938.

Mr. Kirby became a member of the Technical Association of the Pulp and Paper Industry in 1947 and, at the time of his death, was Treasurer of the TAPPI New England Section.

Frank William Egan

Frank W. Egan, founder of the paper and plastic machinery company in Somerville, N. J., bearing his name died on July 29, 1959, in Point Pleasant Hospital, Point Pleasant, N. J., after suffering a stroke. Mr. Egan lived in Brielle, N. J.

Mr. Egan was born in Jersey City, N. J. on Jan. 30, 1892, and attended Columbia University and Rensselaer Polytechnic Institute. His early experience was that of a design engineer for Metal & Thermit Co., Mutual Chemical Co., Borden Co., Frederic Taylor, and Johns-Manville Co.

Frank W. Egan

He joined the J. O. Ross Engineering Corp., New York, N. Y., in 1926 as a sales engineer and remained with this company and its associated company, the John Waldron Corp., New Brunswick, N. J., until 1946 when he founded Frank W. Egan & Co. in Somerville, N. J., with his two sons.

Mr. Egan was considered one of the foremost authorities in the country on paper coating and laminating and published numerous articles on the subject. He became a member of the Technical Association of the Pulp and Paper Industry in 1928 and was a former chairman of its Coating Committee.

Mr. Egan is survived by his wife, Mrs. Louise Reynolds Egan, two sons, Edward F. and Lawrence W. Egan, both of Martinsville, N. J., and six grandchildren.

Lewis Gregory Neubauer

Lewis Gregory Neubauer, Chief Chemist of Tasman Pulp and Paper Co. Ltd., Kawerau, New Zealand, with his wife, Mrs. G. Neubauer, were killed under very tragic circumstances in a road accident on July 18, 1959.

Dr. Neubauer was born in Greymouth on Sept. 7, 1916, and was educated at Greymouth Main School and Greymouth Technical College. In 1938 he graduated B.Sc. and in 1939 M.Sc with first class honours in Chemistry at Victoria University, Wellington. He was awarded the Jacob Joseph Scholarship in 1939.

His earlier work was in the field of organic chemistry as a member of the staff of the Dominion Laboratory, Wellington. During the period from 1936 to 1946 while with the Dominion Laboratory Dr. Neubauer carried out toxicological work for several years with studies of the fiber of *Phormium Tenax*, the alkaloid content of ragwort and of other plant products and the resins and other extractives of New Zealand timbers.

Awarded a National Research Fellowship in 1946 he undertook a two-year postgraduate study of wood and cellulose chemistry at McGill University, Canada, where he obtained the degree of Ph.D. While in North America and with particular reference to the establishment of the Murupara Scheme for the forest utilization in New Zealand he spent several months in the Southland Paper Mills at Lufkin, Tex., and visited Canadian and American Universities and other laboratories engaged in the study of wood chemistry and many mills engaged in the production of kraft and newsprint.

Returning to New Zealand in 1950 he rejoined the Dominion Laboratory and later became Section Head of the Organic and Wood Chemistry Section of the Laboratory working mainly on the structural components of wood. In 1955 he joined the staff of Tasman Pulp and Paper Co. Limited as Chief Chemist and established the Technical Control Department of the company.

Dr. Neubauer was a Fellow of the New Zealand Institute of Chemistry and a member of the Technical Association of the Pulp and Paper Industry, Technical Section of the Canadian Pulp and Paper Association, Technical Section British Paper and Board Makers' Association, Australian Pulp and Paper Industry Technical Association. His publications included papers on miro resin, activated carbon from char, the pulp and paper industry in New Zealand, and the distribution of barbiturates in the human body.

Dr. Neubauer is survived by his father, Mr. L. Neubauer. A memorial service was held on Wednesday, July 22, in the Church of England, Kawerau. He was a member of the Technical Association of the Pulp and Paper Industry since 1949.

Leonard Smidth

Leonard Smidth, consultant, died suddenly on July 20, 1959, at Purdys, N. Y.

Dr. Smidth was born in Schulenberg, Tex., on July 4, 1899, and received his Ph.D. from Columbia University in 1925.

From 1928 to 1940 Dr. Smidth worked as a research chemist or technical director for Aldur Corp., Glycolite Corp., Geo. A. Lippincott Co., Luco Products Corp., Freeport Sulphur Co., Oval and Wood Dish Co.

In 1940 he joined the Sylvania Industrial Corp. Sylvan Plastics Inc., a subsidiary of Sylvania Industrial Corp. was organized in 1941, principally to develop and exploit the inventions and products of Dr. Smidth. He is said to have been the first person in the United States to have supplied commercially the so-called "straight urea molding material" (called Aldur).

After the entrance of American Cyanamid Co. and Plaskon Co. into the urea resin field, Dr. Smidth returned to consulting work. Sylvan Plastics, Inc. is now a wholly-owned subsidiary of American Viscose Corp.

Dr. Smidth who lived in Wallingford, Pa., is survived by his wife, Elizabeth, his mother, and a brother in Houston, Tex.

Braxton M. Ryan

Braxton M. (Jack) Ryan, senior sales representative of the Asten-Hill Mfg. Co. for the past 30 years, died of a heart attack on Aug. 26, 1959.

Mr. Ryan represented Asten-Hill in the southern states and made his home in New Orleans. He lived at 114 Witham David Parkway, Metairie 20, La.

ENGINEERING DIVISION

Committee Contributions

Questions and Answers

(*18*) *Question:* Some manufacturers are beginning to use aluminum bus in their equipment. What are the latest developments in protecting this material against high resistance or corroded joints in paper mill atmospheres?

(*18.1*) *Answer:* Aluminum bus is finding an ever increasing use in electrical equipments as manufacturers take advantage of its light weight and shaping abilities. With the use of quality silver plating of aluminum and the proper attention to joint makeup, it will give a satisfactory performance in switchgear equipment application, equal to that of other conducting metals.

Aluminum bus in equipments, supplied for use in pulp and paper industries, will give its best service when consideration is given to the environment in which the equipment will be installed and operated. This fundamental applies to all electrical equipment and is directly under the control of the plant layout engineer. The equipment should be installed as far away from the chemically laden air as possible and the air in the vicinity of the equipment should be as dry as possible. The normal load current will generally heat the buses and associated joints high enough to prevent corrosive attack. However, when the equipment is de-energized for prolonged periods it is advisable to install heaters in the equipment. These heaters are available as standard accessories in most manufacturer's equipments. They should be in operation at all times during the de-energization of the equipment and should not be thermostatically controlled. The heat from the load and/or heaters will help prevent condensation on the bus work. It is extremely important to prevent condensation as it, plus the corrosive salts, will attack metal. The relative humidity should be limited to less than 30%.

Designers of the equipment, and plant engineers in the field, have a common problem. All electrical joints are assembled by man power and the human factors are important in obtaining good joints. The workmen must apply proper cleaning techniques and bolt pressures in applying electrical joints; it is a well-known fact that the range of understanding of the problem, the thoroughness and experience of the workman varies considerably. Joint qualities vary likewise. Temperature and chemical content of atmospheres are additional variables often beyond the control of the design engineer and the plant engineer.

The effects of the aforementioned variables are minimized when the contact faces of joints are coated with a quality electro deposit of silver.

A superior silver plating method has been developed for aluminum bus to enable a good electrical joint to be made with the use of standard bolting techniques. Equipments utilizing this method have given outstanding service in the field since 1954, in various locations including paper mills. It is essential that the silver plate be of a high quality, have good adhesion characteristics, and a porosity approaching zero. This is under the control of the manufacturer.

To protect the silver plate from corrosive attack it is recommended that a protective compound (grease) be applied to all silver plated area. The best of these compounds include a limited amount of zinc chromate, the quantity of which is controlled so as not to affect the conductivity of the joint. The joint is made with the compound on the bus.

Proper application of the equipment, proper silver plating, and the use of bus joint compound will assure a good electrical joint.

The ideal construction would do away with joints completely and have one continuous conductor. A properly welded joint approaches this. Two methods have been developed for welding aluminum and both use an inert gas shielding envelope around the electric arc. In place of flux either helium or an argon gas shield envelopes the welding area and prevents air from forming oxidides with the molten aluminum. The resultant weld gives a low resistance, high strength joint. The welding process is generally confined to joints assembled in the manufacturer's premises. Several equipments are in operation with this type of construction.

In summation: aluminum bus with properly silver plated joint surface is well suited to switchgear equipment because: (1) Inherently the equipment protects the bus from many destructive elements that open busways (particularly outdoor types) are subject to daily, (2) bus temperatures in equipments are normally higher than in the surrounding air and assure a low relative humidity in the immediate bus areas, (3) quality silver plating of aluminum is available from electrical manufacturers, which when used with bus joint compound will result in consistent efficient joints.

High quality control and rigid inspection standards by the manufacturer in silver plating and by the plant engineer and manufacturer in supervising bus joint make-up is the best assurance of satisfactory operation.

(*18.2*) *Answer:* The plating or nonplating of aluminum joints is somewhat a controversial subject. One aluminum manufacturer recommended plating of joints and actually sells preplated aluminum bus while another advocates the use of bare aluminum and pushes the use of a nonoxide compound for the joints.

Switchgear manufacturers in general are strong advocates of silver plating of all joints and connections regardless of whether it is copper and aluminum. In other words, they, as a general rule, do not use nonoxide compounds in factory construction.

Aluminum can be silver plated and presilver plated bus can be obtained from aluminum manufacturers. It is understood that presilver plated bus can be drilled and cut after plating with no adverse effects. Since aluminum bus is silver plated the corrosion and the electrolytic characteristics are the same as with silver plated copper. Therefore, we can see no problem existing.

By using a high-strength aluminum alloy for bus the problem of reduced cold flow is the joints and connections do not loosen due to the heat cycle.

(*22*) *Question:* In paper mills, certain locations experience widely varying temperatures which can cause a change in the strain gage resistance and a differential in the expansion of the member and the expansion of the gage material. The question is, "How can this temperature effect be compensated?"

(*22.1*) *Answer:* The temperature effect may be compensated by the installation of a second strain gage sometimes called a dummy gage. However, care must be taken so that the compensating gage is in the proper circuit of the Wheatstone bridge; otherwise, it will double the effect of the thermally caused resistance changes.

(*25*) *Question:* What precautions must be taken in terminating interlocked armor cables in pulp and paper mills?

(*25.1*) *Answer:* The following precautions have proved valuable in terminating interlocked armor cables in pulp and paper mills:

Within ventilated control rooms or in dry locations.—Power and control cables can be terminated with suitable clamp terminators of the unfilled type. The jacket over the armor should be cut back beyond the clamping point so that the terminator becomes a part of the ground return path.

In the Mill area or in wet locations.—Power cables can be terminated with compound filled clamp-type terminators at the motor terminal box or other end point. Terminator manufacturer's instructions for compound filling should be strictly followed. If at all possible the cable should *not* be terminated into the top of the motor terminal box or other enclosure. Using side or bottom entrance will guard against the possibility of water entering the enclosure. The over-all jacket, if used, should be treated as in dry locations for ground continuity. A further precaution is to paint over the exposed armor with a polyvinyl-chloride compound.

Control cables, if rubber insulated, can be terminated with compound filled clamp type terminators if in extremely damp locations or a transition to conduit can be made before entering the wet area. Otherwise, unfilled clamp type terminators can be used if the precaution of terminating in the vertical plane is observed.

(*26*) *Question:* Where is the best place to measure the pH of white water from the wet end of a paper machine?

(*26.1*) *Answer:* The ideal place is in the tray, or wire pit, beneath the wire. However, the cost of maintaining pH electrodes at this location is usually prohibitive. The electrodes may be mounted in the tray collector at the side of the machine. Another possible location is in a sample line from the primary fan pump discharge. The "best" location must be individually selected for each machine.

Progress in Alkaline Pulping—1954

J. R. BYRNE and D. J. MACLAURIN

An annual review of the technical and topical literature reporting progress in alkaline pulping was prepared for 1946 through 1953 (*136–143*) at The Institute of Paper Chemistry under the joint and partial sponsorship of the *Southern Pulp and Paper Manufacturer* and the Alkaline Pulping Committee of TAPPI. This annual review is now continued by The Institute of Paper Chemistry. It is planned to publish the series for 1954 through 1958 in quick succession, and then maintain the annual review thereafter on a current basis. Each review will report on the literature as abstracted for that year in the *Abstract Bulletin* of The Institute of Paper Chemistry.

EXPANSION, MODERNIZATION, NEW MILLS

United States

Chemical Engineering (*1*) has listed the pulp and paper mills which were planned, under construction, or completed during 1953. *Pulp & Paper* (*2*) gives brief descriptions of eight mills which are scheduled to begin production in 1954. *Pulp & Paper* and other publications (*3, 4, 5*) give a detailed description of the new kraft pulp and board mill of National Container Corp. at Valdosta, Ga., which is designed for a minimum output of 500 tons of containerboard per day. The new 86,000-ton dissolving pulp mill of Rayonier at Jesup, Ga., is described in *Paper Trade Journal* and other publications (*6–10*). The mill uses a significantly new process, having elements of the kraft process but with extensive modifications. Details are not yet ready for publication. Moon (*11*) gives a detailed description of the technical installations at the new Bowaters newsprint mill at Calhoun, Tenn. It was designed initially for the annual production of 125,000 tons of newsprint and 50,000 tons of either unbleached or bleached kraft pulp. A detailed description of the new 300 ton per day dissolving pulp mill at Foley, Fla., of Buckeye Cellulose Corp., a subsidiary of Procter & Gamble of Cincinnati, is given by Evans and others (*12–15*). The mill employs an acid prehydrolysis-kraft process for cooking pinewood, principally longleaf, slash, and loblolly. *Pulp & Paper* (*16*) and Harris (*17*) described the Everett, Wash., bleached sulphate mill of the Weyerhaeuser Timber Co. Chapman (*18*) reported on the start-up problems incurred at Everett, and Alcorn (*19*) discussed the engineering of the mill including the reasons for constructing the mill, selection of site, and planning for the required facilities. *Pulp & Paper* (*20*) reported on the recent expansion of the Springfield, Ore., Pulp Division of the Weyerhaeuser Timber Co., which increased kraft containerboard production from 250 tons to 350 tons per day. Completion of a $5,000,000 expansion program of the St. Regis Kraft Center in Pensacola, Fla. (*21*), and completion of a new kraft mill for St. Regis at Jacksonville, Fla. (*22*), are reported. *Paper Mill News* (*23*) reports the completion of a $4,000,000 expansion and modernization program at the P. H. Glatfelter Co. of Spring Grove, Pa. Important features of the program were the conversion from the soda process to the sulphate process, and an increase in daily production of bleached pulp from 120 to 160 tons. The new four-stage hardwood kraft bleach plant of Oxford Paper Co. at Rumford, Me., is described briefly (*24*). Cassell (*25*) gives a rather detailed description of the changes made at the Everett Pulp and Paper Co. A 50% increase in chemical pulp and about 30% increase in machine output was accomplished. *Paper Trade Journal* (*26*) describes the latest features of the program of continued expansion and modernization at the Savannah, Ga., plant of Union Bag and Paper Co. Recent additions to the kraft mill include six digesters, a new evaporator, and a black liquor recovery unit. Evans (*27*) describes the rejuvenation of the D. M. Bare Paper Co., Roaring Springs, Pa., a subsidiary of the Combined Locks Paper Co., Combined Locks, Wis.

Canada

Brown and Macey (*28*) give a description of the operations and equipment of the mill at Port Mellon of Canadian Forest Products Ltd., resulting from an extensive modernization program. The potential productive capacity of the mill is now about 200 tons per day of dry, unbleached kraft pulp. *Pulp and Paper Magazine of Canada* (*29*) gives illustrations showing the construction of a new sulphate mill building and some of the equipment installed at Irving Pulp & Paper Ltd., St. John, N. B.

Europe

The modernization program of the Waterhouse Paper Mills of Henry Cooke & Co., Ltd., Beetham, England, is described (*30*). A new kraft paper mill at Kotka, Finland, has been completed (*31*). A brief description of the new straw-pulp mill, La Cellulose de Bigny in France, is given (*32*). Its output is expected to reach 500 metric tons per month. *Paper Industry* (*33*) describes by means of illustrations the reconstruction of the obsolete sulphate mill at Frantschach, Austria.

South Africa

Paper Maker (*London*) (*34*) reports that the new mill of South African Pulp & Paper Industries, Ltd., at Mandini, is ready to start up.

Israel

Paper Market and other publications (*35*) describe the recently opened plant of the American-Israeli Paper Mills, Ltd., at Hadera, Israel. The mill will produce kraft, newsprint, and groundwood-content and groundwood-free writing and printing papers.

J. R. BYRNE, Research Assistant, and D. J. MACLAURIN, Chief, Pulping and Papermaking Section, The Institute of Paper Chemistry, Appleton, Wis.

New Zealand

The kraft paper mill of New Zealand Forest Products Ltd. at Kinleith is described (*36–38*). This is the first kraft mill in New Zealand. Production began in November, 1953, and the mill was officially opened on Feb. 20, 1954.

THE COOKING PROCESS

Kulkarni (*39*) has investigated the physical mechanism of wood pulping by the kraft process. Simkin (*40*) studied the effect of high alkalinity in kraft cooking. Richter and MacClaren (*41*) obtained a patent for a method of preparing wood pulp suitable for conversion to cellulose esters by a two-stage process involving a treatment with an aqueous solution of sulphur dioxide at 100 to 110°C. and subsequent pulping with kraft green liquor. Conversion to white liquor is thus avoided. A Canadian patent was awarded to Sloman (*42*) for a two-stage pulping process using black liquor as the pulping agent in the first stage and white liquor in the second. A saving of 2% caustic is reported in the pulping of eucalyptus wood. A process in which a group of digesters are connected in parallel between liquor supply and return manifolds through which liquor is continuously circulated is the subject of a patent obtained by Westcott and Field (*43*). The digesters are connected so that as each digester completes the cooking schedule it may be blown, refilled with chips, and returned to the system without interfering with the operation of the other units. The liquor returning to the digesters is sampled and fortified with fresh liquor as required. Pulp of a more uniform quality, as well as a more uniform steam demand, is claimed for the system. Rozenberger (*44*) gave the theoretical and practical considerations of the rapid kraft cooking process. Among the variables studied were amounts of heartwood and sapwood, time, temperature, chip size, and preimpregnation of the chips. McKee (*45*) gives an outline of the principle and procedures of the hydrotropic pulping process with sodium xylenesulphonate and emphasizes its advantages over the standard sulphate process. A continuous cooking developed by Kamyr for the production of high-quality kraft and semichemical pulps is outlined by Lundberg (*46*). The advantages claimed for the process (reduced digester corrosion, low chemical and power consumption, absolute control of cooking temperature and time, and pulps of superior quality) are discussed. Nolan (*47*) went on with his work in continuous alkaline pulping. Continuous methods are compared with industrial batch methods from the standpoint of chemical usage and recovery. A series of equations is developed by which the evaporation loads may be calculated for each system in combination with different washing systems. Hart (*48*) describes the applications of the Va-Purge process to alkaline and sulphite batch systems and continuous equipment. The Va-Purge process is a pretreatment technique for the removal of both surrounding and included air by means of a steam-purging treatment involving pressure cycles. Vorreiter (*49*) reports on the lignogen process, a new method for obtaining a long-fibered pulp from barked or unbarked healthy wood waste. The raw material is cut into blocks and cooked with an alkaline liquor (exact composition not disclosed). The pulp occupies an intermediate position between fine wood excelsior and kraft pulp and is best used for felts or boards. Centola (*50*) has pointed out that mixtures of very dissimilar woods can be cooked when autohydrolysis (aqueous prehydrolysis) followed by a sulphate cook is employed. A very good dissolving pulp is obtained. Even mixtures of softwoods, and hardwoods gave some pulps with excellent solubility and uniform characteristics.

Kosaya (*51*) has investigated the use of birch, aspen, and spruce separately and mixed in the manufacture of kraft pulp. Bhat and Virmani (*52*) report that bleached sulphate pulps in satisfactory yield on a laboratory scale were obtained from two species of wattlewood. The large wattle plantations in India raised for tanning bark should make considerable quantities of the wood available soon as a by-product. Chidester (*53*) reviews the suitability of the different pulping processes to the pulping of hardwoods. All conventional processes offer possibilities, but the kraft and semichemical processes are at present of greatest interest. Gruen (*54*) presented data on the extent of hardwood use in the New England area. The sulphate and soda processes have been the largest users of hardwoods in that region. Bergek (*55*) discussed the characteristics of birch pulps and possible uses. Bleached sulphate birch pulp is suitable for writing and printing papers for which bulk and opacity are more important than strength. On the other hand, it is not suitable for kraft paper. Jensen and Bruun (*56*) carried out comparative sulphate cooks with birchwood from different parts of Finland. Wood from the southern and central parts of the country was found to give better yields and strength properties than wood from the northern part. Jensen, *et al.* (*57*), studied the relationship between the viscosity and strength of bleached birch sulphate and sulphite pulps. The pulps were bleached to alter the viscosities and beaten in a Lampén mill. The data indicate that the strength values are fairly independent of the degree of polymerization (D.P.) down to a viscosity of 35 to 40 cp.

MacLaurin and Whalen (*58*) prepared a range of high-yield (60 to 70%) Douglas-fir kraft pulps from lumber waste. The better pulps had strength properties about equal to those obtained from typical average corrugating medium pulps. Jeffreys (*59*) discussed the pulping of eucalypts in Australia. The Australian eucalypts will not produce kraft pulps with strength properties as high as those made with eucalypts from other parts of the world, because in Australia most of the pulpwood is drawn from mature and overmature forests, and has probably grown at a much slower rate.

Merewether (*60*) studied the effect of alkaline pulping on the lignin of *Eucalyptus regnans*. The analysis of the alkali lignin derivatives and those formed from lignin isolated by ethanolysis suggests that alkaline pulping of *Eucalyptus regnans* results in the elimination of two alcoholic hydroxyl groups and a carbonyl group. Bland and Batterham (*61*) have investigated the progress of delignification of the wood of *Eucalyptus regnans F. Muell.* with sodium hydroxide and sodium sulphide at various temperatures and concentrations. The action of sodium hydroxide speeded up consider-

ably with an increase in temperature. The sulphide acted more rapidly than the hydroxide, but the effect of temperature was not so great, although increase in concentration accelerated the action of the sulphide more than that of the hydroxide. Custis (*62*) reported on a complete sodium balance made at the Camas kraft mill to determine with some accuracy the sodium losses from the three major sources of loss which are furnace stack gases, the pulp leaving the mill, and the main sewer. Expressed as pounds of sodium sulphate per ton of air-dry pulp, furnace stack losses were 20 per 25 lb., pulp soda losses 40 to 45 lb., and sewer losses 30 lbs. Bogomolov (*63*) studied three sulphur compounds formed during kraft cooking. They were hydrogen sulphide, methyl mercaptan, and dimethyl sulphide. Enkvist (*64*) showed that solid thiolignins with high sulphur content are slowly formed in sprucewood by hydrosulphide ions or by thiosulphate ions at pH 7 and 100°C., and possibly also in the first stages of a common sulphate digestion. Later in sulphate cooking the thiolignins are dissolved, split off sulphur, are gradually changed to common sulphate lignin containing only about 2% sulphur. Experiments seem to show that phenyl carbinol groups or the corresponding dialkyl ethers can easily react with hydrosulphide ions, but only if the benzene nucleus contains an activating free phenolic hydroxy group. Schwerin (*65*) describes work which was done to determine the suitability of cupriethylenediamine as a solvent for alkali-cooked pulps, and to establish the approximate viscosity range of these pulps. The solvent proved reasonably stable and measurements were reproducible and sensitive to small structural differences of the pulps. Samuelson and Wennerblom (*66*) investigated the degradation of cotton cellulose during cooking with alkali at 170°C. The mechanism and kinetics of degradation of cellulose by alkaline cooking with the formation of carboxyl groups are discussed. Brink (*67*) studied the effect of aqueous solutions of pure sodium hydrosulphide on the constituents of wood and found it cleaves protolignin linkages in aspenwood. Because hydrogen sulphide is formed, repolymerization of the cleavage products is minimized as opposed to the normal kraft cook where repolymerization occurs under the strongly alkaline conditions. Mattson (*68*) examined recycled black liquor used as a diluent in preparing sulphate cooking liquors, and found that the organic components of the black liquor are relatively unaffected by recooking. These components affect the pulping process, however, in that the resulting pulps have increased permanganate numbers and lignin contents and decreased brightnesses and alpha-cellulose contents.

WASHING AND SCREENING

Gehm and Moggio (*69*) reported on water conservation in the kraft pulp and paper industry. Water usage per ton of product has declined steadily in the past 20 years. Present-day mills can operate successfully using 20,000 to 30,000 gal. of water per ton of unbleached pulp. The basic factors bringing about the decrease in water consumption are the reuse of process water, new types of equipment, and improvements in economies in manufacturing operations. West and Bergstrom (*70*) describe the new brown stock pulp washing system at the Thilmany Pulp and Paper Co., Kaukauna, Wis. Armstrong (*71*) reviewed the merits and drawbacks of diffuser, vacuum, and press washing. Particular emphasis is given to press washing, and its application to high-yield kraft pulp is mentioned. Calhoun (*72*) described the process of continuous countercurrent brown-stock washing on vacuum filters. Foam control is emphasized. Ranhagen (*73*) discusses modern Swedish practices and developments in the screening of sulphate pulp. Ratliff (*74*) discusses the installation of a Lindblad high-intensity screening system at Gaylord Container Corp.'s Bogalusa, La., sulphate pulp mill. A maximum capacity of 500 tons of air-dry pulp per day was obtained through the four Lindblad screens connected as planned. Homans and Pineo (*75*) reported on trial runs of a high-density screening system for high-quality bleached kraft pulp. The main objective of the trial was the evaluation of the productive capacity of the high-density system in relation to the conventional flat-screen installation. Under proper stock consistency control, it appears that the high-density system would have many advantages over the flat-screen system. Gardner, *et al.* (*76*), reports that early experience with Cowan screens on kraft pulp was not satisfactory. This experience was obtained with 0.085-in. perforations on 0.125-in. isometric centers. Later, tests were made with 0.085-in. perforations on 0.1445-in, isometric centers which showed greatly improved performance.

SULPHATE LIQUOR CYCLE

Hatch (*77*) outlines the basic theories underlying the different steps in the kraft recovery process, and discusses the unavoidable losses in kraft recovery. Pineo (*78*) discusses the functions of the chemical recovery system in kraft mills and factors affecting the ratio of sodium to sulphur compounds. A brief outline is presented by Gibson (*79*) of the six steps in the recovery system and discusses some of the usual control tests used in the different stages. A Jenkins Bros. advertisement (*80*) describes the piping layout for a black liquor recovery system. Kennedy (*81*) describes kraft recovery instrumentation by following the spent cooking liquor through the various stages. Smelik and Habiger (*82*) discuss the use of antimony electrodes in the titration of dark liquors. Prior to determining sodium hydroxide, sodium carbonate, and organically bound sodium, hydrogen peroxide is added to destroy the negatively charged sulphur. Then the antimony electrode establishes several equivalence points mentioned in the article. Chêne and Martin-Borret (*83*) compared three different methods of determining free caustic soda in spent liquors. A conductometric titration using diluted liquor and a potentiometric procedure carried out with undiluted liquor gave identical results. The Michel-Jaffard and Nicollet technique gave results from 9.8 to 12.5% lower than those given by the other two. The error is possibly due to the fact that a large excess of barium chloride in ethanol is required which causes flocculation of organic compounds which, in turn, results in occlusion of sodium hydroxide in the flocculation. Hougberg and Enkvist (*84*) found that in the determination of hydrosulphide, sulphite, and thiosulphate ions in kraft black liquors using standard titration methods, it is necessary to protect the liquors against the in-

fluence of oxygen in the air which oxidizes the hydrosulphide ions to thiosulphate ions very rapidly. Bergstrom (*85*) gave brief extracts from more comprehensive investigations on by-products obtained during the cooking of kraft pulp. Adler and Hagglund (*86*) have solubilized lignin in black liquor by treatment with an oxidizing gas, then with a sulphonating agent and an aldehyde at temperatures of 100 to 160°C. Following a review of the development of black liquor oxidation and the pertinent literature in the field, Tahtinen (*87*) discussed the influence of black liquor oxidation on the heat economy in alkaline recovery and its advantages and disadvantages. Aries and Pollak (*88*) describe a method of oxidizing sodium sulphide from waste liquors to sodium sulphite by mixing the liquor with sodium carbonate at temperatures of 275 to 295°F. in the presence of an excess of air. Gray, *et al.* (*89*), obtained a patent for a treatment of spent cooking liquors in which the dissolved smelt after combustion is alternately carbonated under pressure and steam-stripped in a partial vacuum which removes hydrogen sulphide. Two other patents (*90*, *91*) of a similar nature were also issued to these men. Owens (*92*) described the purpose, design features, functions, and efficiency of the recovery unit and discussed its importance for the pulp mill and power plant. Bergstrom and Waters (*93*) outlined possible operating troubles and their prevention or correction in kraft black liquor evaporation. Armacost (*94*) obtained a patent for an evaporation system for black liquors which incorporates a spray tower into which the liquor is introduced at 30 to 40% solids and from which the liquor is pumped to the furnace burner unit. A soundwave generator is mounted in the top of the tower and is claimed to afford intimate mixing of the liquor with the hot gases rising through the tower. Nolan and Kulkarni (*95*) show the black liquor evaporation loads which might be expected when continuous methods of pulping are used to replace the batch methods now in use. Stoess (*96*) describes a pneumatic method for conveying the make-up salt cake to the mixing bin which eliminates the present service bin, screw feeder, and vibrating screen. A filter screen for use on concentrated black liquor before spraying into the recovery furnace is described by Hochmuth (*97*). Gamble (*98*) reports on the recovery furnace and boiler unit installed recently at the Karnaphuli Paper Mills at Chandraghona, East Pakistan. The unit has a capacity of 65 tons of soda per day and produces 30,000 lbs of steam per hour at 425 p.s.i. and 775°F. *Pulp & Paper* (*99*) reports on a new No. 14 Combustion Engineering recovery boiler which went into operation recently at Longview Fibre Co., Longview, Wash. Baker (*100*) discusses the heat input and distribution in pulping a regular grade of kraft pulp. For each ton of air-dried pulp produced, 11,730 lbs. of steam at 212°F. are available from the burning of black liquor; of this quantity, 2100 lb of steam are not used in the pulping process. Greenawalt (*101*, *102*) obtained an American and Canadian patent for a furnace for the recovery of heat and chemicals from a soda process or similar spent liquor which is lined with coils through which liquid coolant is circulated to control the temperature within the combustion chamber. Bories (*103*) presents calculations to compare the amounts of heat and power which may be recovered from black liquors fed to the furnaces with 55, 70, and 90% dry solids. Hanssen (*104*) presents an historical review of various methods for utilizing the heat value of spent cooking liquors. He emphasizes that for certain cases, wet combustion seems to offer advantages over evaporation and combustion, such as higher heat economy, safe operation, and smaller investments. Bauer and Dorland (*105*) present and discuss equilibrium constants for some reactions involved in the combustion of sodium-base pulping liquors. Reasons for sulphur and sodium losses are pointed out. Aries and Pollak (*106*) obtained a patent for a process in which smelt from the burning of black liquors is dissolved in a circulating concentrated sodium sulphide solution to which water is added as the concentrated solution is withdrawn. This solution has a limited solubility for sodium carbonate and sodium sulphate, which settle out in crystalline form. Brown (*107*) discusses three types of deposits found in green liquor systems, their causes, and corrective measures that could be taken. Gery (*108*) reports on a survey of 27 southern pulp mills followed by a 4-year program of research, development, and field tests in recausticizing. As a direct result of these studies, a new green liquor clarifier and dregs washer will be incorporated in the standard Dorr causticizing system. Venemark (*109*) shows the use for a rapid, easily reproducible, and accurate routine method for the analysis of white and green liquors in sulphate mills.

STREAM AND AIR POLLUTION

The National Council for Steam Improvement issued its 1953 report (*110*) in which the activities and developments during the past year in sulphite, kraft, semichemical, white water, deinking, and strawboard effluents, stream analysis, aquatic biology, and dehydration of hydrogels are reviewed. *Pulp & Paper* (*111*) describes the system of holding back waste water from April to October and releasing the waste under controlled conditions from October to April. This is being done at the Springhill, La., kraft pulp and paper mill of International Paper Co. MacIntire (*112*) obtained a patent for a treatment in which calcium chloride and orthophosphoric acid are added to alkaline waste water. Dibasic calcium phosphate is formed in the presence of suspended organic matter, and said organic matter is occluded in the process and is precipitated out with the calcium phosphate. The process claimed in another patent by MacIntire (*113*) is similar to the one described above except that standard superphosphate is substituted for the orthophosphoric acid. The subject of a patent by Zimmerman (*114*) is the oxidation of waste effluents by the introduction of air under pressure into a tank containing the waste material heated to the point at which oxidation will take place. Heat recovery from the unit is claimed to be 95% of the theoretical value in the material. Bishop (*115*) reports that, as a result of the work of the National Council for Stream Improvement, Southland Paper Mills has installed a full mill-scale treatment plant employing the aeration process for accelerated B.O.D. reduction of kraft mill wastes.

Von Bergen (*116*) discusses the possible serious consequences of lack of attention to the public relations

aspect of air pollution, with particular reference to kraft mills. Zirm (*117*) gave a brief summary of recent work done by E. I. du Pont de Nemours & Co. on industrial deodorants for the kraft industry. Kusian (*118*) discusses briefly the properties of odors and methods of odor measurement and reviews present odor control procedures. Tremaine (*119*) reports on the use of masking agents on odor nuisance problems, with particular reference to kraft mills. Ghisoni (*120*) describes the air pollution problems of the sulphate mill of Cartiera Vita Mayer & Co., in Italy, which is situated in a narrow valley.

BLEACHING

A description of the bleach plant at the sulphate mill at Riegelwood, N. C., is given by Dailey (*121*). The application of a high-density chlorine dioxide stage to hardwood and softwood sulphate pulps as the sixth stage of the process is outlined. Pineo (*122*) reviews the present status of sulphate bleaching, covering in some detail the five basic stages and a sixth stage with chlorine dioxide or peroxide. Fennell (*123*) found that in the multistage bleaching of hardwood or softwood kraft pulps where a hypochlorite bleaching agent is followed by alkaline peroxide, the brightness of the final product is improved if the pulp is subjected to acidifying conditions for a short time. Chadeyron (*124*) discusses the difficulties in measuring the brightness of wet pulps and also discusses the effects of heat and ultraviolet light on the brightness of two types of kraft pulps and two types of sulphite pulps. Aitken (*125*) reports that sulphate pulps can be bleached to brightnesses of 85 to 90 through the use of sodium peroxide while retaining the same strength obtained with normal bleaching agents at much lower brightnesses. McEwen and Sheldon (*126*) discuss the use of hydrogen peroxide as a final stage treatment in bleaching kraft and soda pulp giving particular attention to moderate density bleaching, poststorage tower bleaching, and to high-density steep bleaching. Schroeder and Seferian (*127*) discuss the improvement in bleach response of sodium peroxide by means of an acid posttreatment to the preceding hypochlorite stage. With proper use of this process, a bleached kraft pulp can be produced in the mid-80's with superior brightness stability and strength properties.

DIGESTER CORROSION

A plea is made by Pesch (*128*) for the inclusion in TAPPI Standards T 624 m-44 and T 625 m-48 of those components which may influence corrosion, particularly polysulphides. Baisch and Whitney (*129*) have presented an annotated bibliography of alkaline digester corrosion covering articles published in this field through the year 1953. Following a review of the factors which have an influence on kraft-digester corrosion, Stockman and Ruus (*130*) present a report about some of the recent investigations carried out by the Swedish Digester Corrosion Committee. Cooking time and temperature and the amount of oxygen in the digester have effects on corrosion. The copper and aluminum content of steels have no influence on corrosion. Shoumatoff and Teeple (*131*) give a detailed report of an industry-wide study of corrosion in alkaline digesters made by numerical analysis of over 37,000 wall thickness measurements in 89 digesters in 12 different mills. Christiansen and Lathrop (*132*) present a progress report of the project initiated at the request of the Alkaline Pulping Committee of the Technical Section of CPPA concerning corrosion in alkaline pulping equipment. Ungar and Caywood (*133*) report on the analysis of pertinent data concerning corrosion in alkaline pulp digesters which have been submitted on 198 digesters by 29 mills to the Technical Association of the Pulp and Paper Industry. Thomas (*134*) presents the history and service record of 100 carbon-brick linings which have been installed in alkaline pulp digesters in North America during the last 22 years. Thin linings have not been so successful as the thicker ones, and their success has been dependent on several factors, which are discussed. Scheil (*135*) reported on some of the conclusions reached by the A. O. Smith Corp. on the potential performance and corrosion rates of various types of stainless steels and alloys in kraft digester service. The need for re-examination of the techniques for obtaining reliable corrosion rates in testing is stressed.

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LETTERS TO THE EDITOR

Photo Micrographs of Paper Surfaces

To the Editor, *Tappi:*

The June, 1959, issue of *Tappi* contains three extremely interesting articles with illustrations of photomicrographs of paper surfaces taken with low angle illumination. I would like to point out that in certain instances these pictures can be very misleading, particularly in the case of coated papers and boards. These pictures are optical illusions which can be interpreted only if the observer has some bias (such as by past experience in viewing surfaces) or if he knows the direction of the incident light. A mountain viewed from above with light incident from the right-hand side looks identical to a chasm with light incident from the left-hand side. A quite astonishing transformation in surface character takes place if one turns a photograph upside down. For this reason it is very desirable to have all photographs appearing in an article with the incident light from the same side and with the light direction labeled. Even when one knows the direction of the light it is often difficult to obtain the correct interpretation. I think it might be helpful to introduce a bias into the picture such as by placing a very small bead on the surface in the section photographed. For example, in the June, 1959, issue of *Tappi*, photographs on pages 487, 488, and 489 all have the light incident from the top of the page, whereas Fig. 11 on page 490 has the light incident from the bottom of the page. In the article appearing in the February, 1959, issue of *Tappi* the top photograph of Fig. 10 has the light incident from the bottom of the photograph and the bottom photograph of the figure has the light incident from the top.

I think that attention to this problem will allow these photomicrographs to attain their full potential value to the reader.

KEITH E. BRADWAY
Union Bag-Camp Paper Corp.
Franklin, Va.

Mannose in Cellulose and Pulps

To the Editor, *Tappi:*

Due to my mistake, an unrevised version of the paper "Mannose in Cellulose and Pulps" by Matsuzaki, Ward, and Murray, appeared on page 474 of *Tappi* for June, 1959. As a result, discussion of previous work is incomplete and one table has been omitted. Table V should appear at the end of the paper.

The following discussion should appear at the end of the second paragraph on page 475.

Table V. Nitrogen Content of Cotton

Boll	*Material absorbed*	*Nitrogen content, %*
I-I	None (control)	0.04
III-I	Mannose	0.04
V-1	Glucosamine hydrochloride	0.00
V-3	*N*-acetylglucosamine	0.03

"However, since glucose alone gives a spot under similar conditions, it is not presently possible to interpret these results. The spots in question are probably the same as those observed by Saeman, Moore, Mitchell, and Millett [*Tappi* 37:336 (1954)] when glucose was subjected to their standard procedure for hydrolyzing cellulose, since our hot dilute acid treatment is the same as their second stage.

Roudier and Eberhard [*Mém. sérv. chim. état* 40:261 (1955)] noticed epimerization of acid solutions of sugars which were boiled during neutralization with barium carbonate, but this may be due to the difficulty of getting hydroxide-free carbonate. Of course, our deionizing resins may also have had localized alkaline regions, but we consider this unlikely since no spots were observed in experiments 13 and 14 (Table III) using the same resin.

As Speck points out in his review on page 63, Volume XIII, of "Advances in Carbohydrate Chemistry," acid epimerization of sugars is known. However, only fructose has been identified from the treatment of glucose with sulfuric acid [*Ost. Agnew. Chem.* 18: 1170 (1905)]. Mannose has not been identified from this reaction."

In this same paragraph, second on page 475, the second sentence should begin "Xylose has been independently identified. . .," and the correct reference is to Das, Mitra, and Wareham, *Nature* 174: 1058 (1954).

It may be well to mention something in connection with another paper "Lead Tetraacetate Oxidation of Pulps and Hydrolyzed Pulps," by Matsuzaki and Ward on page 516 of the same issue. A paper by Roudier and Nick [*Mém. serv. chim état* 41: 181 (1957)] has only now become available to us. This investigation is similar to our own and our findings corroborate theirs very well. I regret that we did not know of this paper at the time of our publication.

Kyle Ward, Jr.
Senior Research Associate
The Institute of Paper Chemistry
Appleton, Wis.

RECENT BOOKS

Forest Fire: Control and Use. By K. P. Davis, G. M. Bryan, and W. R. Krumm. McGraw-Hill Book Co. Inc., New York, 1959. Cloth, 6 × 9, 584 pages. $12.

The voracious consumption of natural resources has given an urgency to the development of more efficient methods and practices for their conservation. New methods will undoubtedly allow a more economical use of resources, but in the case of forests, a constant battle must be waged to curb the depredations of fires. Although the brunt of the responsibility for the control of fire is shouldered by trained personnel, the public is also able to share this responsibility and thus greatly decrease the incidence of wanton or accidental destruction. Indeed, the more information the public possesses, of the inestimable riches of forests and the destruction wrought by forest fires, the more alert will it be in guarding against a regrettable negligence.

The present book contains material which would interest both the specialist and the layman. The causes, control, and effects of fires are discussed at length. The Maine Fire of 1947, e.g., was caused by "blow-up" burning conditions (drought, high air temperature, and relative humidity) and the presence of untended fires. In contrast, the friction in power-log skidding, together with an incendiary, was responsible for the Tillamook Fire of Oregon in 1933; over 300,000 acres were destroyed in this fire. Beside burning fuel, the heat effects accompanying forest fires are also injurious; they kill or injure vegetation and animal life, alter the character of the soil by producing residual mineral products, and often are more serious than the mere consumption of wood. Many eyewitness accounts of fire-fighting are also given.

Technometrics. Edited by J. Stuart Hunter. Published quarterly by the American Society for Quality Control and the American Statistical Association, Wm. Byrd Press, Richmond, Va. Annual subscription rate: members, $6.00; nonmembers, $8.00.

A new journal of statistics of interest to mathematicians, experimental and natural scientists, it is published under the joint sponsorship of the ASQC and the ASA. Articles describe new statistical techniques helpful in the physical sciences, illustrate the application of known statistical methods in new or novel environments, offer tuition on particular statistical methods, and deal with the philosophy and problems of experimentation and quality control. Further information about the journal can be obtained from either sponsor.

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Automatic Refining Control

DONALD P. MICHEL

AUTOMATIC refining control has a ready-made niche for it in our paper industry. The requirements in practically all new equipment are for more precise automatic controls rather than for the handwheel type of adjustment. Papermaking still may be considered an art but more profit-conscious pulp and paper mills are relying on instruments instead of the taste, smell, and feel of the operator.

By automatic control, whether it be on a refiner, digester, jordan, or paper machine, we mean the continuous sensing of variables and the resultant correction or compensation to maintain the variables within preset limits.

The paper industry as a whole has made great strides in the use of automatic controls, especially in the last 4 to 5 years. Credit for these strides can be laid to research for better technology, new and improved equipment, a deeper scientific look at the art of papermaking, and the help and understanding of the various committees of TAPPI. Certainly among the motivating factors were the greater and more exacting demands of paper products and increased production costs.

Frankly, we would like to know what the future expects of us and our equipment. Of one thing we can be sure: automatic controlling devices are here to stay. You have told us that. Many of your problems on quality, uniformity, etc., in the pulp mill or paper mill could be solved today if we put together: the proper understanding of the problem, the knowledge of the available instruments, and the willingness to adapt to different or new techniques. It is generally this willingness that makes a new innovation work.

Other industries can teach us much. For example, take a look at the instrumentation and control systems used in the petroleum and chemical industries. These industries have gone through the same growing pains as the paper industry. Today they are employing large digital computers to optimize their production and quality. It is our opinion that the pulp and paper field will be utilizing such type of control within the very near future. In order to profit by new techniques we must continually reappraise the entire instrumentation and control program for the paper industry.

As new developments in other industries are revealed, we should check for applications in our own industry. Today, we have many examples of the nuclear, electronic, and aircraft contributions to new types of controls for the paper industry. Beta ray gages, electronically-controlled machine drives, electric hydraulic actuators, digital and analog computers are a few of the recent developments that have been adapted. Proper tailoring of these and other new ideas to our own problems can help advance the day of the "perfect" pulp and paper mill.

Through much of the basic and applied research to date, many of the mysteries of papermaking have been unraveled to the point where automatic or computer control is feasible for complete refining systems and entire pulp and paper mills. Such a mill or system could use temperatures, pressures, flows, consistency, freeness, and the actual finished sheet characteristics as feedback loops. Data on current market conditions, products, quality or quantity, labor and material costs could be used as additional inputs. We could expect that such a mill would produce the correct grade, quantity, and uniform quality for the least cost at the right time for the best market.

With the future in mind, we are designing today, control systems on individual machines that could be integrated with the complete plant control systems of tomorrow. Our couch vacuum control is another contribution in that direction.

DONALD P. MICHEL, Electronics Engineer, The Bauer Bros. Co., Springfield, Ohio.

APPLICATION

Our first commercial installation of the couch vacuum control system was at Otsego Falls Paper Mills, Otsego, Mich. This mill produces 9-pt. semichemical corrugating medium on the two paper machines. The stock flow for each machine prior to the Bauer installation was as shown in Fig. 1. The

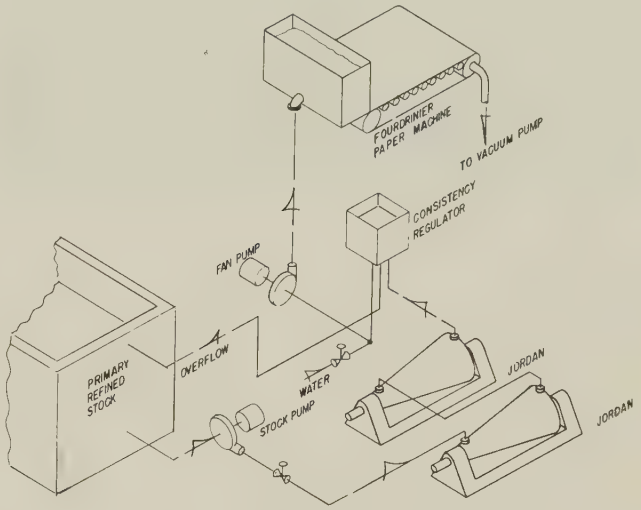

Fig. 1. Original equipment layout, Otsego Falls Paper Mill, Otsego, Mich.

primary refined stock was pumped through two jordans in series to the consistency regulator with a small overflow. The regulated stock dropped into the fan pump and then to the headbox.

The Bauer no. 442 was put into the line in place of one of the jordans (see Fig. 2). A vacuum recorder controller with high-low set points and a sampling circuit to reduce the possibilities of the system hunting or overcontrolling was installed and wired to the hydraulic system of the refiner.

On start-up in the spring of 1956, the system was put on automatic control. Despite the changes of primary stock freeness, the refiner was able to keep full control. Records of both couch vacuum and refiner hydraulic closing pressure show changes of the refiner in response to the couch vacuum (Fig. 3). The hydraulic pressure in this refiner is directly proportioned to the amount of work being done by the refiner.

The control points on the vacuum recorder-controller were set so that a variation of $\pm 1/2$ in. of vacuum would send a correction signal to the refiner. This signal would make a small hydraulic adjustment which, in turn, would change

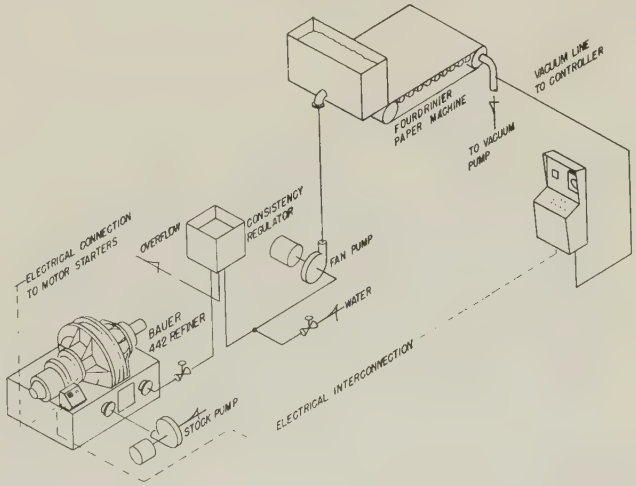

Fig. 2. Present equipment layout, Otsego Falls Paper Mill, Otsego, Mich.

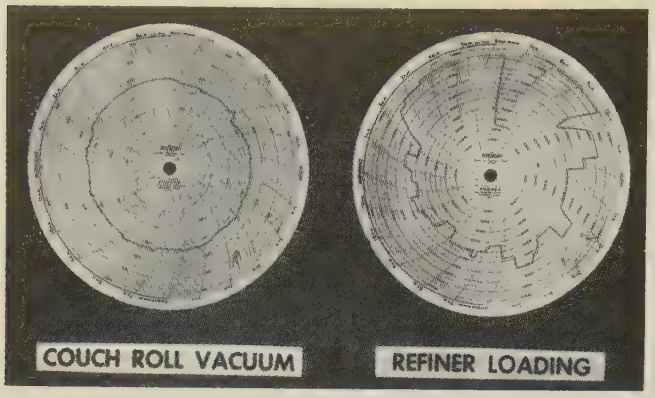

Fig. 3

the amount of work being done by the refiner. A timer would then keep the correction circuit inactive for a period of time. The length of this pause is rather critical as will be pointed out later. It is set equal to or slightly greater than the time that the stock takes to go from the refiner to the couch roll.

Sheet quality could vary up to 25% before the change-over to the Bauer no. 442 couch vacuum system. With the new system the quality can be maintained within 2% variation and sheet tests have been increased.

Several other mills are using this same system on the larger Bauer pump-through refiners with very good success. One mill has set the high and low limits at $\pm 1/4$-in. vacuum with the result that a much closer control of sheet quality is maintained.

In mills where tonnages require two or more refiners in parallel to process the stock, the couch vacuum control would still be applicable. Both refiners would respond to the signal in the same manner but an automatic override to prevent one refiner from taking all the load would be used. Such a system has been offered recently to several large southern mills.

THEORY

The operating principle of the couch vacuum system is that a change of couch vacuum indicates a change of sheet formation. Sheet formation is dependent upon the amount of stock preparation. Where the couch roll is not used, other indications are available to the machine tender to tell that the stock is of the proper quality. In the case as described by this paper, the vacuum variations are used by many to indicate a change of sheet quality. Therefore, by using this information and allowing it to reset the refining equipment, the control of sheet formation is automatically accomplished.

The Bauer no. 440 and no. 442 pump-through refiners are high capacity, slow speed, single revolving disk refiners.

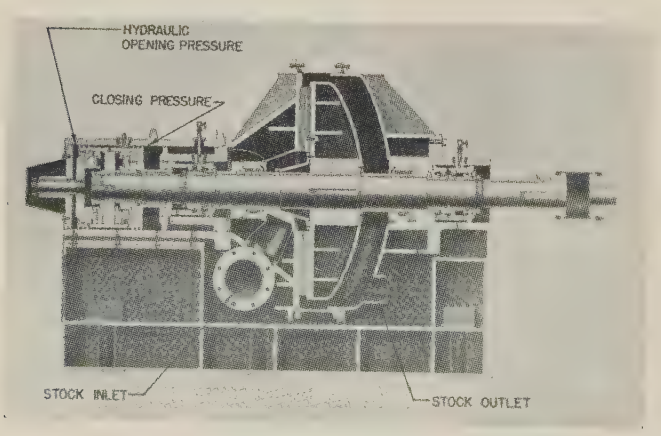

Fig. 4. Bauer 440 pump through refiner

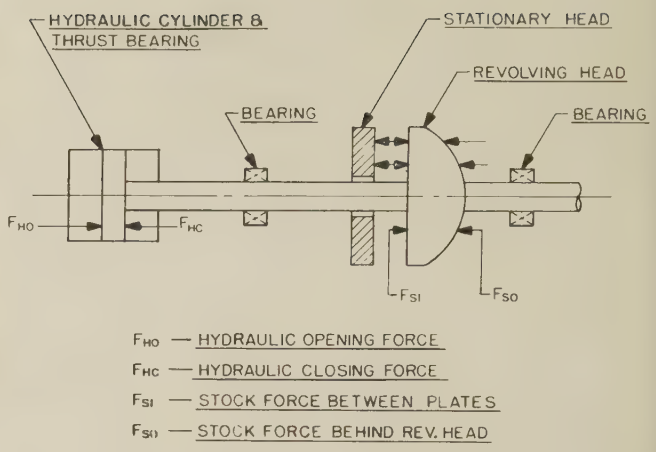

Fig. 5. Bauer 440 hydraulic schematic

These refiners have been used in a wide variety of applications in stock preparation fields. The fact that the refiners are hydraulically loaded makes them ideally suited to an automatic operation using signals from the couch vacuum system. Other control signals also could be used to regulate the operation of these refiners.

Basically the refiner consists of a stationary head, a rotating disk mounted on a through shaft, a hydraulic cylinder and a hydraulic system. Figure 4 shows a cut-away of the larger pump-through refiner. In Fig. 5 a schematic representation of the refiner shows the hydraulic forces which position the refining disks. In this diagram F_{si} is the pressure of the stock to the refiner plus the pressure necessary to produce the refining action on the stock. Force F_{so} is the hydraulic force built up in the case and acting on the back of the disk due to the action of the plates. This force can range from zero to a value greater than the stock pressure F_{si}.

Force F_{ho} is the hydraulic opening force. Most generally this force is a constant and is necessary to permit control where the pumping action of the plates produces a high value of F_{so} and a low value of F_{hc}. The hydraulic force F_{hc} is the closing force and is varied to position the refining heads. An equation for these forces under most operating conditions would be $F_{si} - F_{so} = F_{ho} - F_{hc}$.

In most applications the stock pressure into the refiner will remain constant, as will the hydraulic opening force F_{ho}. These two forces tend to increase the distance between the refining plates. The stock pressure on the back of the revolving head and the hydraulic closing pressure will add together to bring the two to a steady condition. Further changes in the hydraulic closing force F_{hc} will change the loading of the refiner driving motor by increasing the pressure on the stock between the plates. This loading is proportional then to the hydraulic closing force and the refiner plate pattern.

The vacuum recorder controller in each installation has been a standard instrument. The scale range of the instrument is 0 to 30 in. of mercury. A run of $1/4$-in. tubing is made between the instrument and a point in the suction

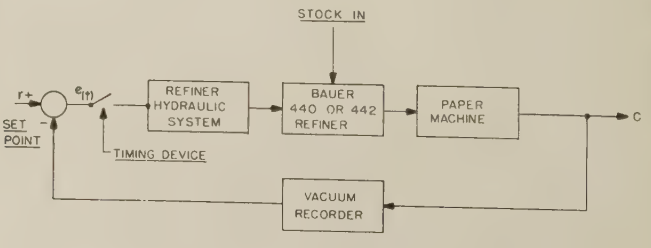

Fig. 6. Block diagram couch vacuum control system

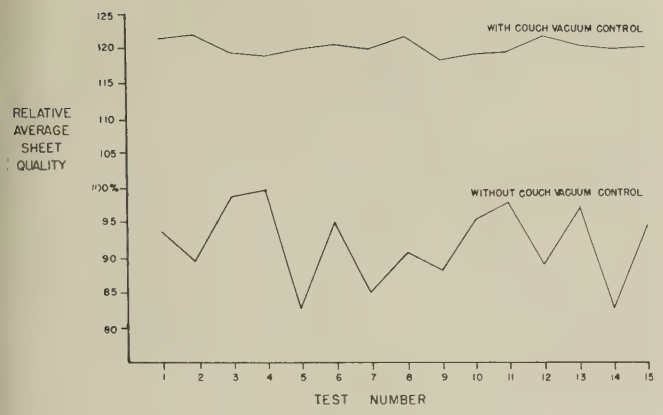

Fig. 7

pipe close to the couch roll. Vibration dampeners in the form of snubbers, orifices, or a long run of small tubing may be used to filter out high frequency pen excursions. A band width for this control of from 0 to 5 c.p.s. is sufficient to pass the information that we are seeking.

The instrument has a single knob set point which positions both the high and low limits of operation. For best response the "dead" zone between the high and low limits should be 1 in. of vacuum or less. Too small a dead zone with a pen which has a rather high rate of oscillation would cause the control system to be continually operating. Therefore, pen vibration and dead zone are rather critical factors.

The control circuit consists of two timers which operate only when a vacuum limit has been exceeded. The first timer makes a small change to the hydraulic closing pressure on the refiner. The second timer then holds the entire circuit open until the newly refined stock has had a chance to pass the couch roll. Obviously, if this time were too short, hunting could occur. If the time were too long, the system would have poor response.

A block diagram (see Fig. 6) shows a little more clearly how these individual elements fit together. With this type of diagram, the application of closed-loop theory is a relatively easy matter. The three blocks of paper machine, Bauer pump-through refiner and the hydraulic system can be lumped into a single block with an over-all gain of G.

A rigorous methematical analysis of the stability criteria for this type of sample-data control system is given in John Truxal's book, "Control Systems Synthesis."

CONCLUSIONS

One paper mill superintendent has been quoted as saying that the Bauer automatic refining control is a papermaker's dream. The reasons he gave were ease of operation, quick response to changes in stock, and a better and more uniform sheet which means fewer breaks, better drying, more machine speed, and better machine press action.

The benefits of the automatic system are important to the industry. So important, that equipment manufacturers must consider them as criteria in designing their new machinery.

The pulp and paper industry is moving toward completely automatic control with the eventual possibility of computer supervision for the entire operation. The Bauer automatic refining control is one step toward the realization of that goal.

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2. Truxall, John G., "Automatic Feedback Control System Synthesis," New York McGraw-Hill.

Presented at the Thirteenth Engineering Conference of the Technical Association of the Pulp and Paper Industry, Portland, Ore., July 28–Aug. 1, 1958.

Application of Newer Flash Drying Techniques to High Grade Chemical Pulps

T. W. DUNBAR

Flash drying, or pneumatic conveying drying, is a method of drying whereby the material is dispersed into small particles and introduced into a hot gas stream which conveys and drys these particles. The hot gases are separated from the medium to be dried by means of a centrifugal collector. John Breakey Ltd., in Breakeyville, Que., have had a 100-ton Glomera flash drier in continuous operation for approximately 11 months on the drying of groundwood pulp, and this unit has met with sufficient success to warrant some investigation of the drying of high grade chemical pulps by this method.

Because the pneumatic conveying type of drying is an entirely new concept in the pulp and paper industry, it is necessary to consider the dewatering and baling sections in addition to the drying portion itself, as conventional methods do not necessarily satisfy the requirements of this type of drying. During this discussion we will, therefore, include the dewatering and baling sections in our discussions on flash drying.

PILOT PLANT TESTS

Using the pilot plant which was originally used to develop the Glomera flash system as installed at John Breakey Ltd., some tests have been made on sulphate pulp in Vancouver, B. C. This pilot plant consists of a pulp fluffer, a Glomera drying tower, a 25-gal. oil burner, an end fired horizontal cylindrical furnace, and two separating cyclones. In addition to this single-stage drying unit, we have modified an existing hydraulic press to compress pulp into sheets of varying densities.

Approximately 3000 moisture-free pounds of unbleached sulphate pulp were flash dried in our pilot plant, and the same pulp was dried on a machine using cylinder driers followed by a Flakt drier section. Both these samples were subjected to full beater tests, including measurement of bulk, and the results are shown in Table I.

The flash-dried pulp exhibited a low burst factor and tear factor, but a high bulk in the unbeaten condition. After the flash-dried pulp had been beaten for 40 min. to a freeness of 300, the burst was normal, but the tear and bulk were slightly above normal levels. The flash-dried pulp, when made into handsheets, appeared to be distinctly lighter than that of the regular-dried pulp, however no specific tests were made to confirm this. The flash dried pulp proved definitely easier to reslush than the pulp which had been dried on a machine using cylinders and a Flakt drier in series.

Subsequent tests were made on 80 G.E. bleached sulphate pulp after being pressed through a screw press to a moisture content of 56.5% and also after being flash-dried to a moisture content of 10.8%. The G.E. brightness before drying was 77.8 and after drying was 77.5. None of this same batch of pulp was dried by other means, so the other tests on this sample were not significant.

Considerable additional testing is required on the effects of flash drying on sulphate pulps, however, the above results, together with the present degree of acceptance on the groundwood flash dried pulp, are sufficient to warrant an additional investigation.

MECHANICAL DEWATERING

In flash drying techniques, a sheet is not required before drying. In fact, there are advantages in not having a sheet form, as if the sheet is pressed to any degree, this only en-

T. W. Dunbar, President, D. & S. Engineering Ltd., Vancouver, B. C.

hances the difficulty of fluffing the material to be dried. With this in mind, a screw press was originally tried at John Breakey Ltd., as the most efficient means of dewatering

Table I. Test Results on Unbleached Sulphate

Time	*Burst*	*Tear*	*Freeness*	*Bulk*
		Regular Dried		
0	21.9	223	697	1.622
30	71.8	124	456	1.316
60	81.1	112	106	1.215
45	76.5	117	300	...
		Flash Dried		
0	10.2	182	697	1.983
30	71.3	147	385	1.353
60	86.9	126	115	1.245
40	77.0	140	300	...

up to a point where the material could be fluffed and flash dried. These tests on groundwood, and subsequent tests on chemical pulps, indicate that this type of press in its present development creates knots or rolled up fibers which are unsatisfactory in the finished product.

A disk type of press known as the P & L dewatering press, many of which had been in use in the food industry for some time, was tested as a pulp dewatering unit. The press consists of two stainless steel perforated disks covered with stainless steel screens rotated by a variable speed driving unit. The material to be pressed is fed in the top where the disks are furthest apart, and is pulled with the disks into the narrow space toward the bottom of the press, with the water being extruded through the screens and disks. The pulp is then discharged in a cake form from the top of the press. In order to feed the pulp into the press in sufficiently dry condition, at John Breakey Ltd. a standard vacuum type thickener was used to thicken the stock before it was fed into the P & L press.

Extensive tests made this month indicate that by using a special variable speed force feed, sulphate pulp can be dewatered from 3 to 28% air dry in one press, and from 3 to 48% air dry with two P & L presses in series, the force feed being required on the first section only. Each 5-ft. diam. dewatering press has a capacity of 50 tons air dry per 24 hr., therefore a total of four P & L presses, together with two force feed units, are required to dewater 100 air-dry tons per 24 hr. from 3 to 48% air dry. The above mentioned tests showed an average fiber loss of 1 lb. per 1000 gal. Flash drying can be effectively employed after a conventional dewatering section, and in Norway a 100-ton Glomera flash drying unit is being installed after a Kamyr machine which dewaters to approximately 55% air-dry consistency. In this case, the sheet is broken up by spiked rolls before going into the fluffer, whereas the discharge from a P & L or screw press can be discharged directly into the fluffer.

DRYING SECTION

As a source for the hot gases, a direct fired air heater is utilized, burning as a fuel either bunker C fuel oil or natural gas, depending on fuel cost factors. This air heater can be located either horizontally or vertically, depending on available space, and is designed for outdoor installation. Two fans are employed, one to supply the combustion air and the other to provide the necessary quench air, which is mixed with the air heater gases at the discharge from the air heater to provide a constant gas temperature of 400 to 600° F., depending on the type and initial dryness of the pulp being handled. The quench air is forced between the outer casing of the air heater and the inner brick section in order to keep the refractory cool and cut down furnace losses. Unlike a conventional boiler furnace, the direct-fired air heater requires large quantities of excess air to provide the low temperature exit gases required. This allows the use of more excess air than is normally employed through the burner register which, together with a high temperature refractory chamber, provides completely smoke and soot-free operation.

A source of heat for the hot gases could be obtained from a high temperature steam coil, an indirect fired air heater, or an air heater installed in an existing boiler, as local plant conditions deem these alternates economically feasible. We have had no indication to date that there is any color reversion or change in quality of the pulp in any way due to direct firing.

The fluffing is performed with a Novorotor hammermill, consisting of two high-speed disks which operate in opposite directions, and which are fitted with swinging hammers. This fluffing of the pulp into fine particles is very important in flash drying, as fine fluffing or fiberizing makes it possible to dry the product without case hardening or overdrying.

The material is partially dried in the first stage drying section, which consists of a conveying fan, pipe, and discharge cyclone. Because of the higher temperature used, and the fact that the material is wetter in the first stage, it is not necessary to use a tower, as sufficient retention time is obtained with the pipe only.

The second stage consists of a fan and low velocity uplift-type Glomera tower, together with a cyclone. The tower is approximately 60 ft. high, and gives the necessary retention time to permit the required heat transfer between the particles and the hot gases. The last stage of the Glomera pulp drier as installed in John Breakey Ltd., consists of a fan and tower, together with cyclone, the same as the second stage except for the lower temperature employed. During the entire drying cycle, the material is in no lasting contact with the hot metal, avoiding any charring or roasting effects, which might take place. The multistage drying method employed provides low temperature drying during the last portion of the drying cycle, when the pulp is most sensitive to heat, and provides for very accurate control of the moisture content of the finished product.

The pressure in the plenum chamber is controlled by actuating the quench fan damper. The total heat liberated is controlled by the outlet temperature on the no. 1 stage cyclone, where the largest portion of the drying takes place. The temperature of the gases in nos. 2 and 3 stages is controlled by the outlet gas temperature, which actuates louvres, admitting dilution air. The dampers from the plenum chamber to the three stages are operated by remote control from the main control panel, and are used to balance the system.

When starting the unit, the dampers between plenum chamber and the three stages are closed, a damper on a short stack connection from the plenum chamber is opened, and the unit is warmed up for about $^1/_2$ hr. before pulp is admitted to the system, to avoid any possibility of contamination of the pulp when starting a cold furnace.

The outlet temperature of the gases varies with outside temperature and humidity, however at John Breakey it usually averages about 145°F. in the first two stages, and 130°F. leaving the last stage. At John Breakey, reports received indicate an evaporation rate of from 1400 to 1500 B.t.u. per lb. of water evaporated. The pulp is discharged into the baling section at a consistency of 90% air dry.

BALING METHODS

Where rail shipment only is anticipated, and especially in the case of relatively small drying units, a low density baler is used, baling the pulp to only 30 lb. per cu. ft., which in many cases gives maximum allowable car loadings. Where water shipments make up the largest proportion of the total

pulp shipped, the high density baler to compress the pulp to 50 lb. per cu. ft. is used.

The low density press is primarily an extruding machine which forces the pulp continuously through a section where the side guides are adjustable to provide the desired back pressure against which the press piston is working. The press installed at John Breakey Ltd. uses a fly wheel and crank drive with a Ward Leonard variable speed electric drive, with a range of 14 to 45 strokes per min., which will produce minimum and maximum capacities of 30 air-dry tons per day and 90 air-dry tons per day, respectively.

Each stroke of the piston produces a sheet approximately 24 by 12 by 1 in. In order the remove the air compressed by the piston, a small high pressure fan pulls air through holes in the press box.

The sheets discharged from the compressor are stacked and formed into bales 24 by 40 in., which weigh approximately 550 lb. each on a modified wet pulp hydraulic baler. These bales are strapped with standard $^3/_4$-in. steel bands for rail car shipment.

In order to cut down initial cost and make the baler somewhat more flexible, the crank and fly wheel drive has been replaced with a hydraulic piston assembly on a new pulp compressor which is presently being built. This pulp compressor has a two-circuit hydraulic system consisting of a high-speed approach cycle, powered by a high capacity low pressure pump working through a regenerative circuit, and a high pressure 5000-lb. low capacity pump completing the final portion of the compression stroke. For high density baling, a completely new type of press was developed. This press is of vertical design. The dried and fluffed pulp is discharged from two cyclones and drops down two chutes, one on each side of the press, where it is converted into bales.

There appear to be advantages to forming the bale in sheets, rather than in a continuous stroke. In addition to having smaller higher speed machine requiring less building and foundation, the pulp breaks up more easily into sheets and absorbs more water more easily in the repulper due to the additional contact area provided by the sheet form.

SYSTEM COSTS

The capital cost of a complete flash drying system, including mechanical dewatering, drying section, and high-density baling system, together with all foundations and installation labor, but not including the building which is required around the dewatering and pressing sections only, varies from $3000 per ton for a 50-ton unit, to $2400 per ton for a 250-ton unit. The total direct operating cost, including labor at $1.50 per hr., fuel at $0.10 per gal., and electricity at $0.07 per kw.-hr. varies from $5.55 per ton for a 50-ton unit to $3.80 per ton for a 250-ton plant.

CONCLUSIONS

Full scale plant operation for almost a year indicates that flash drying is generally satisfactory for groundwood pulp. This operation has allowed for mechanical improvement in flash drying techniques, including mechanical dewatering and baling sections.

Tests on unbleached sulphate indicate that flash drying produces pulp which is comparable in physical characteristics and somewhat easier to repulp than this same pulp dried by conventional methods. Preliminary tests on 80 G.E. bleached sulphate indicates that the flash drying does not affect the brightness appreciably.

Flash drying generally has some obvious advantages over regular drying systems.

1. Lower initial capital cost.
   a. Lower equipment cost.
   b. Drying section proper designed for outdoor location, allowing lower building cost.
   c. Lower installation labor cost.
   d. No steam plant required.
2. Lower operating cost.
   a. Unit designed for operation by one man on driers and two men baling.
   b. Lower maintenance, due to elimination of felts, wires and rotating parts, plus steam plant equipment.
   c. Approximately the same oil consumption per ton.

It would appear that flash drying of sulphate pulps should be considered in any evaluation requiring additional drying capacity. In view of the comparative newness of this type of drying as applied to sulphate pulp, it would seem reasonable to have tests made on the specific pulps required to be dried to make certain that the end results provide the required physical properties when a flash drying plant is contemplated for sulphate pulp.

Presented at the Thirteenth Engineering Conference of the Technical Association of the Pulp and Paper Industry, July 28–Aug. 1, 1958.

The author wishes to thank the management of John Breakey Ltd. and Canadian Sumner Iron Works Ltd. for the information and experience gained through pilot plant and full scale operation of this type of pulp drier. The author wishes to thank Louis Gagnon, mill superintendant at John Breakey Ltd., for his outstanding contributions in the originational design, and development of this drier.

Performance and Operational Characteristics of Worm Gear Drier Drives

W. ERNEST STONE

This paper will of necessity be in the nature of a discussion of the subject rather than a presentation of factual data in support of the various points to be developed. This is due to the relative newness of this type of drier drive and the great difficulty of accumulating data which can be used in setting up "before" and "after" comparisons.

HISTORY

I referred to the "newness" of worm gear drier drives, but I have in mind our present-day approach to this subject. Actually, there are drier parts now in operation with the paper driers in orthodox two-tier arrangement with each drier driven by an individual worm gear set. These are not Rice Barton installations. They are on news machines for the most part and some were installed as far back as 1925. I have seen one of these running at a speed in excess of 2000 f.p.m.—1800 f.p.m. is common. The original design speed was more like 1000 f.p.m.

Despite what appears to us today as obvious deficiencies in the design of the older installations resulting in higher-than-average maintenance costs, the mill people with whom I have talked are pleased with them and believe that the advantages far outweigh the disadvantages. I hear the thought expressed that the same type of drive would be preferred on a new machine.

Rice Barton Corp. has for some time been aware of the plight of many of the smaller mills, particularly in the fine paper grades. These have been operating for years with outmoded equipment, each passing year finding them that much farther behind the times with the same old machines and that much less able to make the capital investment to bring them up to date—a vicious circle. We have given much thought to the matter of developing up-to-date machine features at costs within the reach of paper manufacturers in this class.

One such feature is the worm gear for individual drier drives. This was originally intended to give the paper mills an enclosed gear drive for small machines at a cost appreciably below that of standard enclosed gears. It does just that and apparently much more.

W. Ernest Stone, Sales Manager, Rice Barton Corp., Worcester, Mass.

MECHANICAL DETAILS

Before going into a discussion of the advantages and disadvantages of this system of gearing you may be interested in the construction of these units.

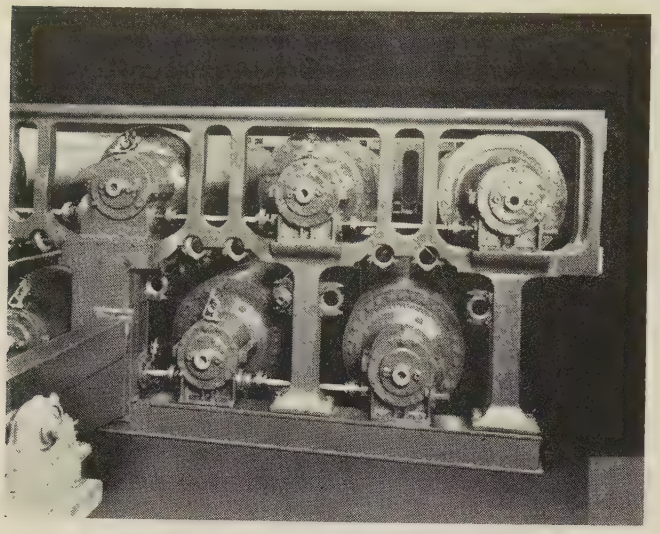

Fig. 1. Drier part—drive side

Figure 1 is typical of our first three installations. It shows part of the drier section erected in our shop before shipment. The felt rolls are not in place. Remember that I referred to these as intended for small machines. They were for 36 and 48 in. driers with faces not exceeding 110. A line shaft extends the length of the drier section at both levels with the worm of each individual drive set into and a part of each line shaft. The connecting portions of these line shafts are floating shafts with flexible couplings. The two line shafts in each section are positively connected so that there is only one power input point in each drier section. A drier part in a standard

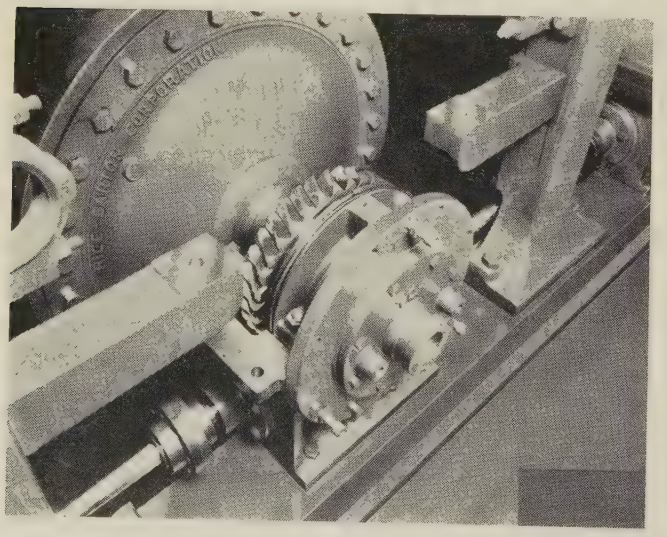

Fig. 2. Worm gear drive unit—single

machine would, of course, be made up of a number of sections. May I call to your attention that the external dimensions of this unit are well within the periphery of even a 36-in. drier so that except for the line shafts there is no more interference with ventilation than on the front side.

The internal construction of each unit is shown in Fig. 2. The gear casing is also the pedestal housing for the drive side antifriction drier bearing and the worm gear is mounted directly on the drier journal. The antifriction worm bearings are carried in this same casing so that once adjusted there can be no misalignment of any of the elements in this assembly. The drive side drier bearing is the "held" bearing so that any axial misalignment of the worm wheel relative to the worm due to temperature changes is reduced to a minimum.

One or two design features I think are worth mentioning. The gear ratio must be sufficiently low to allow coasting when the power is cut-off. The reversal of the power flow through the worm gear under this condition must not be permitted to lock the drive.

The selection of the size of the unit is dictated more by the drier journal diameter than by the actual power requirements. This works out to more than ample capacity. Note in this connection that each worm gear drives only its own drier although the worm itself must have sufficient capacity as a shaft (not in the screw thread) to drive the units which may be beyond.

The initial setup adjustment is made by moving the carriage which carries the drier bearing so that in effect the entire drier is moved slightly to bring the worm wheel in line with the worm. Once assembled neither the drier bearing nor the worm wheel is ever disturbed on the journal except for possible replacement.

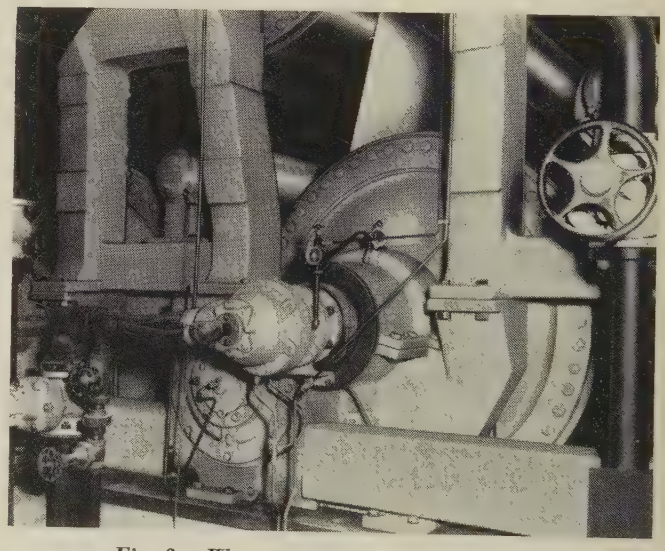

Fig. 3. Worm gear drive unit—double

This unit is designed for ease of maintenance. Complete disassembly can be achieved in place and if necessary the rim of the worm wheel can be replaced over the bearing itself without removing it.

We also have three installations on larger, faster machines—one a 216-in. board machine with 60-in. driers for 1500 f.p.m. Under these conditions the high speed means that the line shaft speed would be higher than we would like to run it. This is one reason for the high maintenance costs mentioned earlier with regard to the pioneer installations. We have, therefore, introduced a set of helical gears in each unit in addition to the worm gear (Fig. 3). These are in reality step-up gears with the smaller or driven helical mounted on the drier journal and the larger or driving gear on a jack shaft with the worm wheel. Any temperature change displacement is taken in the helicals which is a good place for it.

Other features are as described above for the single gear unit. Note that here again the controlling dimensions are within the peripheral limits of 48-in. as well as 60-in. driers.

ADVANTAGES AND DISADVANTAGES

And now we can take up what we gain or lose by this type of drier drive and the performance characteristics.

Previously, I mentioned the price advantage of the single units. The cost actually lies between the usual type of enclosed small diameter helical gear systems and the older large diameter open gears. In the case of the double type, however, we lose our price advantage over the standard enclosed gears as the costs, up to the present at any rate, appear to break even. On the larger machines, advantages other than price make these drives attractive.

Perhaps the main consideration is the matter of ventilation. I don't have to tell you that this is too large a subject to handle in detail here, but I would like to touch on a few factors.

The first of these stems from the point mentioned previously that the front and back sides of the machine are equally open from a ventilation standpoint. The front and back side drier frames are identical and the gear casings offer no obstruction as they are in line with the driers and do not extend beyond the drier faces to impede air flow from or into the "pockets." In other words, ventilationwise the drier part is symmetrical about the machine center line and, neglecting other factors, the sheet should dry symmetrically with good opportunity for better control of unequal drying. At least this unknown variable is eliminated.

In a recent discussion with a mill official who has made a specialty of drying problems, he mentioned an extreme case of this type of trouble. He was called in as an expert to study a serious drying problem on a wide, fast machine with orthodox enclosed gears. This drier part was totally enclosed by the hood. He found drying conditions so different at the front

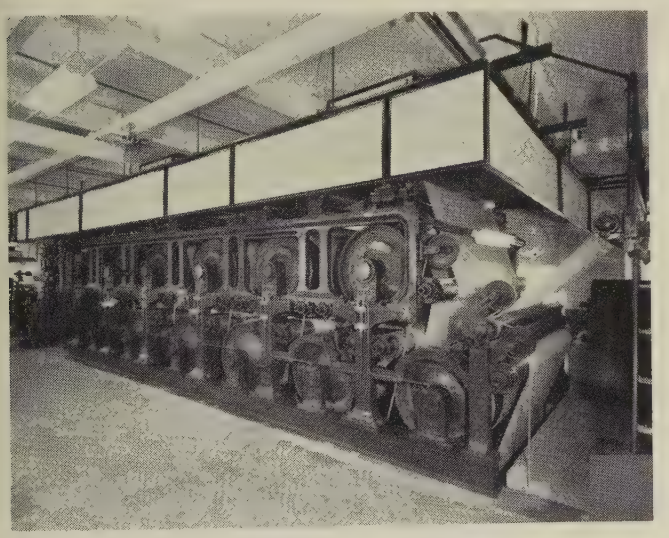

Fig. 4. Drier part—tending side

side as compared to the back that his first move was to baffle the front side to simulate conditions at the back and thus set up a symmetrical drying pattern as the basis for further study.

Another factor in the matter of ventilation is the drier spacing—vertical and horizontal. There is an optimum spacing for every sheet and, of course, an average for a range of grades on one machine. With the worm gear drive, there is no limitation on the relative drier locations set up by the necessity for holding gear centers. With worm gears, horizontal spacing is a matter of length of the connecting section in the line shaft and the vertical spacing is varied merely by changing the length of the vertical connecting shaft. It is a simple matter to adjust the framing dimensions to suit the desired spacing.

We received unexpected benefits in our first installation due to this locational flexibility (Fig. 4). This was a 12-drier, two-deck section in a fine paper mill with eight more driers to be added later to achieve the desired production. We were able to space the driers so that two felt rolls could be used between the driers instead of one. This meant that the sheet and felt contacted and left each drier almost simultaneously. The drier areas with the sheet in contact but not the felt were negligible. Previously, the tendency of the sheet to cockle at these points meant running with low steam pressures with consequent low production. Under the new condition, however, the control of the sheet by the felt made higher steam pressures possible, greatly increasing the production over that anticipated and losing us the sale of the eight additional driers which were no longer needed. Later installations were made, however, on two other machines of this same mill with equally gratifying results.

I cannot promise phenomenal results like this in every case, unfortunately. On wider faster machines the felt rolls must be of such a diameter as to preclude their use in pairs as this would require horizontal spacing wide enough to seriously cut into the sheet contact on the driers.

Data are now being accumulated from several sources to determine if any gain in drying rate can be attributed to the use of worm gear drives, other factors remaining constant.

There are a number of purely mechanical advantages accruing to this design. Among them is the open character of the back side construction. I would not go so far as to suggest that you spear broke from the back side but as far as the drier construction is concerned it could be done. Generally speaking, back side accessibility is greatly improved and is nearly on a par with the front side.

There are also several points in favor of the separate, enclosed, identical gear units in each drier. Only the power required to drive its own drier passes through each worm gear; there is no load from other driers, except through the sheet or felt, and, by the same token, no cumulative backlash.

In the same connection, to mention a less pleasant experience, we failed in our first board machine installation to take account of the very heavy draw going from the drier part into the first calender. As pointed out above, the gears are not self-locking and the power to pull a large part of the drier section passed in reverse through the worm gear set on the last drier. This caused failure of the worm gear hubs in this area of the drier part. Sometimes we learn the hard way!

Even in failure, however, these units have an advantage. As isolated units, a failure in any one gear set is isolated and not communicated immediately and automatically in a chain reaction to the adjacent drier drives as in the case of continuous gear trains. The design incidentally takes into account ease of inspection and replacement if necessary.

Similarly load irregularities in a given drier are not freely transmitted from drier to drier and, therefore, do not build up a synchronized pulsation.

I would like to say a few words also about the quiet operation of a drier part powdered with worm gears. One of the main reasons for discontinuing the use of open gears is the whine and ring of the tooth contacts at high pitch line velocities. The use of composition gears alternating with cast iron has raised the tolerable limit somewhat and a further improvement has been realized using smaller diameter helical gears at lower pitch line velocities totally enclosed and better lubricated.

With worm gear sets, there is the same noise and lubrication advantage of enclosed helical gears plus the inherent gain of the gradual tooth contact. The lower noise level of the worm gears attracts attention immediately in such a machine room.

Presented at the Thirteenth Engineering Conference of the Technical Association of the Pulp and Paper Industry, Portland, Ore., July 28–Aug. 1, 1958.

Mechanical Drives for Paper Machines

OTIS R. WITWORTH

In the past few years, there have been many advances in both the types and range of applicability of mechanical drives for paper machines. At one time, there were ranges of machine speeds and widths that were beyond the capacity of any known mechanical drive and only electrical drives could fulfill the requirements. Today, there are available mechanical drives of a size and accuracy capable of driving the largest and fastest machines. If the need arises larger capacity drives can be made.

Mechanical drives can be divided into two main categories—namely, belt drives and differential drives. The belt drives can be subdivided into flat belt and V-belt types. Also, the flat belt drives can use leather, rubber, or the new nylon core belts.

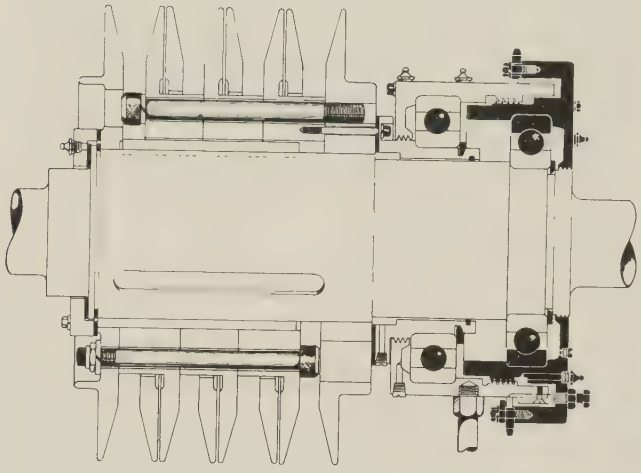

Fig. 1

BELT DRIVES

The belt drives, other than for the necessary changes required for the belts themselves, are similar. Gear boxes can have either the spiral bevel or hypoid types of gears, and either can be compounded with additional gear sets, to get the proper over-all ratio. Both types can be equipped with auxiliary motors for inching over the machine in either the forward or reverse or both directions. Slack take-up devices are also easily included. If it is desired, remote draw indicators can be installed for easy duplication of draw rates. The draw control mechanism can be either hand or electrically operated.

Today, almost every drive is equipped with air operated clutches, but a few are still made with the mechanical engaging devices. The clutches can be either quill mounted or cut-off mounted with each having its own advantages. The quill mounting produces the more compact drive, while the cut-off type is more flexible, from the standpoint of sizes of sheaves, and is more easily adaptable to the higher horsepower ratings.

Flat Belts

The advent of the nylon core belt has put new life into a tried and proved method of driving paper machines. Up to horsepowers of 100 to 125, the leather belt, and somewhat above this value the rubber belt, has been used for many years. As these belts become wider, they are progressively harder to control and, at the same time, require much flatter

Otis R. Witworth, Staff Engineer, The Black-Clawson Co., Inc., Paper Machine Div., Watertown, N. Y.

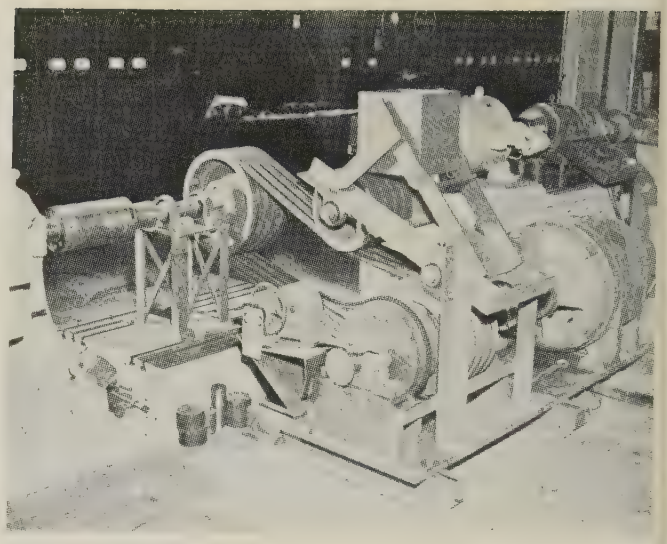

Fig. 2

tapers on the cone pulleys. This leads to a serious limitation to the amount of draw variation, unless extremely wide face pulleys are used. With the nylon core belt, belt widths are reduced to about one third of the width of a leather belt, for equal horsepower ratings, and it is possible to get as high as 340 hp.

At the present time, there are no test data available which indicate the percentage regulation that can be achieved with a properly designed flat belt drive. We do know, for equal horsepower drives, that the narrower nylon belt will track truer and reduce draw variations. In several cases, where trouble was experienced with draw variations, the use of nylon belts corrected the problem. However, until the necessary tests are made, we must assume that the regulation on these drives is not so good as for the other types.

We can also make these nylon belt drives either as short center type, similar to V-belt drives, or as a normal flat belt drive with the lineshaft in the basement or overhead.

V-Belts

The short center V-belt drive has been used for a great number of machine drives. In order to accomplish the necessary draw variations, the Allis-Chalmers Vari-Pitch sheave (see Fig. 1) must be used, which permits a very large draw variation. The variation in draw possible is approximately 17%. It is also possible to transmit as high as 400 hp.

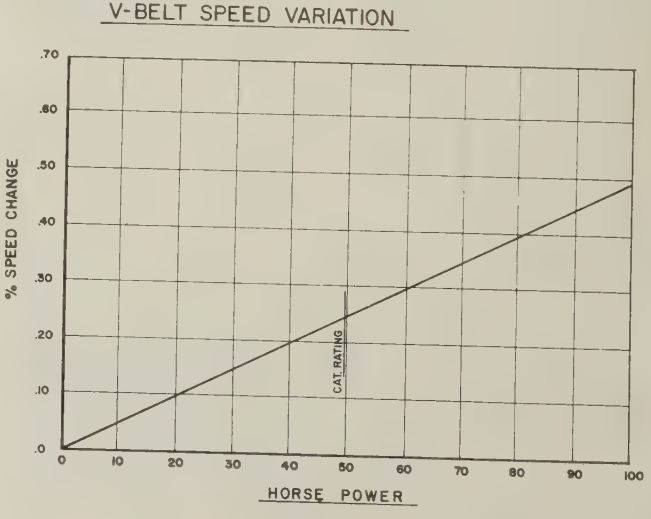

Fig. 3

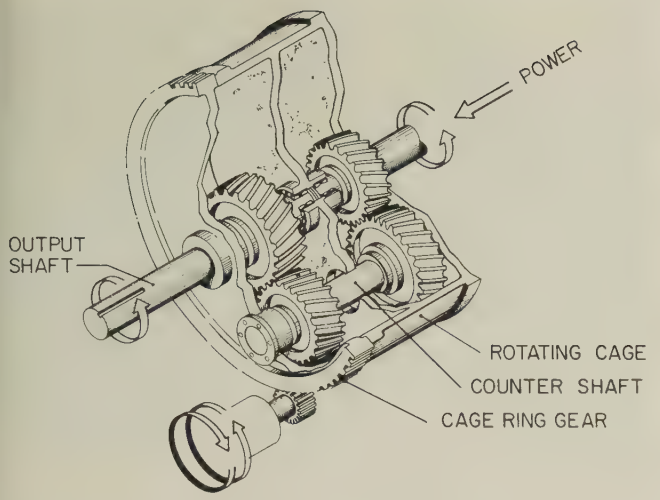

Fig. 4 Differential drive mechanism

The speed regulation of these drives is extremely good and you can realize regulations of 0.16% when properly applied. In order to arrive at these figures, exhaustive tests were made, in conjunction with the Allis-Chalmers Mfg. Co., in which the speed variations were accurately measured as shown in Fig. 2. From the graphs drawn (see Fig. 3), it can be seen that if the belts have a running load of two thirds of the catalog rating, the regulation is 0.16%. With the belts applied in this manner, there is available three times the running load torque to accelerate the machine.

DIFFERENTIAL DRIVES

Since the type of differential drive which we make is new, it would be best to go into some of the details of construction and operation. This drive is patented under pat. no. 2,835,-144.

The differential mechanism is of the helical gear type as shown in Fig. 4. With this type of mechanism, it is not necessary to have bevel or internal gears but it is necessary to have some gear ratio. The power is delivered to the input sun gear, which meshes with a planet gear. This planet gear is rigidly connected to a second planet gear, driving the output sun gear. If we assume that the input sun gear is held, then rotation of the cage, in which the planet gears are mounted, caused the output shaft to rotate in the same direction, at a reduced speed. Rotation of the output shaft can be caused to reverse by reversing the cage rotation.

Function

The manner in which this differential is utilized, in the drive, is as shown in Fig. 5. Power comes in through the lineshaft and is transferred to a jack shaft with a pair of 1

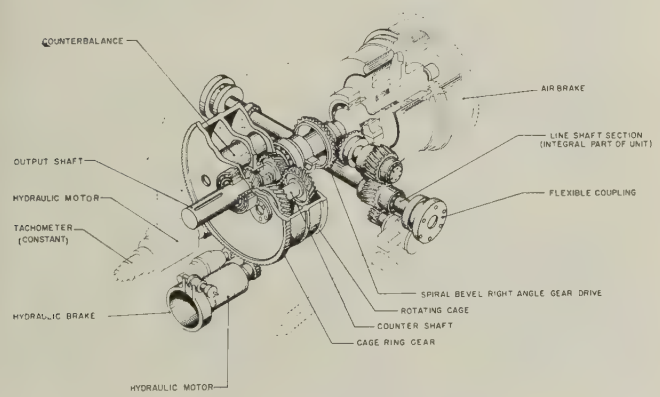

Fig. 5. Differential drive

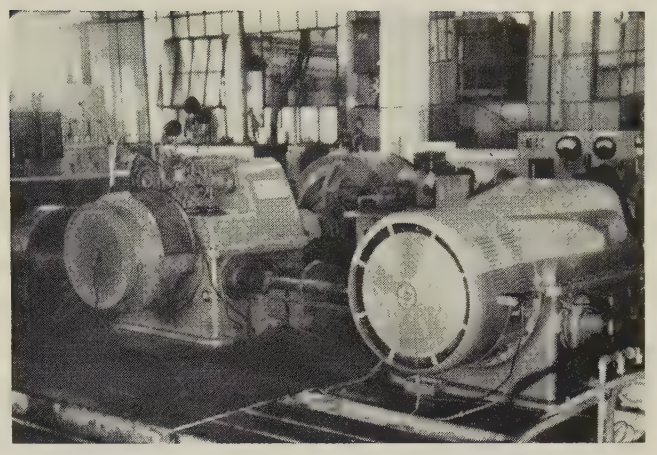

Fig. 6

to 1 ratio gears. Connected to the jack shaft is the pinion of a spiral bevel gear set with a 2 to 1 reduction. The bevel gear is quill mounted and, by means of the clutch, is connectable to the input sun gear shaft in the differential mechanism. Rotation of the cage is accomplished by means of an external ring gear, on the output end of the differential cage, which is driven by hydraulic motor. Also geared to, and driven by, the cage is the speed sensing tachometer which has a fixed displacement. The reference tachometer is located beneath, and is driven by, the line shaft. This tachometer has a variable displacement, which is used to adjust the draw rate.

The hydraulic system, which is used to rotate the differential cage, is a relatively simple circuit and has been used for many applications. A variable displacement pump, driven at constant speed by an electric motor, drives a constant displacement motor at a speed proportional to the output of the pump. This output is set by the movement of a slide block, in the pump, which determines the pump eccentricity or displacement. The tachometer circuit sets the pump eccentricity by means of the pressure which exists, at the time, in the circuit. If we assume that the reference or lineshaft tachometer has been set, by the machine operator, at a specific displacement, this displacement and the lineshaft speed determine the output. This output is fed into the fixed displacement tachometer which is driven by the cage. If the cage is turning slower than it should, the cage tachometer will not take the full output of the lineshaft tachometer and the pressure in the line connecting the two will increase. This increase in pressure will, through a hydraulic cylinder, shift the eccentricity of the pump and change its output. If the pressure in the lines decreases from the cage running too fast, the reverse occurs. The various

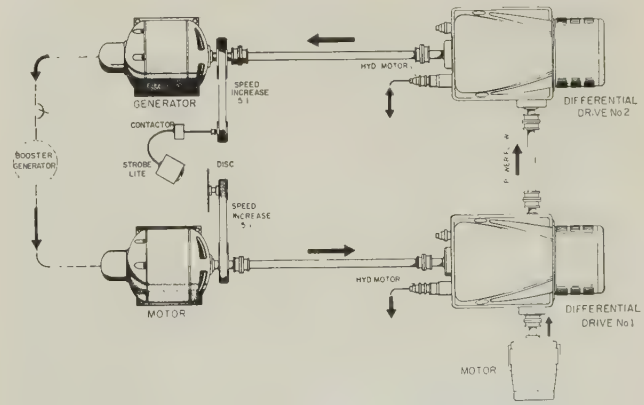

Fig. 7. Principles of differential drive test loading and speed detection

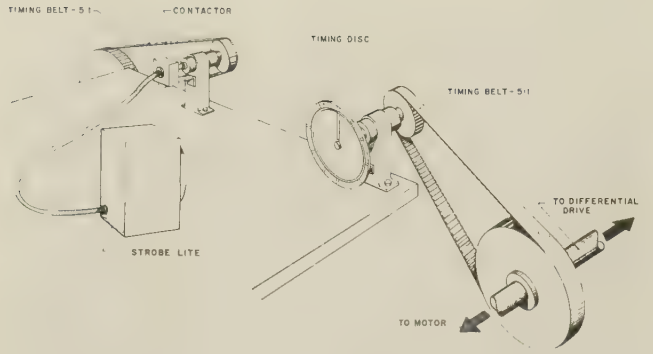

Fig. 8. Differential drive test showing Strobelite setup

functions are secured by inserting valves in the circuit to alter the pressure appropriately.

All of the necessary functions can easily be included with this type of differential drive. These functions include inching forward and reverse and slack take-up. Inching may be used with the lineshaft either running or at a standstill. In order to accomplish any of these functions it is merely necessary to add solenoid valves to the hydraulic pumping units and to install the necessary electrical circuits to operate the solenoids. Electrical interlocks are provided in order to prevent accidentally activating the wrong function.

Specifications

The specifications for these drives are as follows:

1. Speeds—up to 1000 r.p.m. of the lineshaft.
2. Over-all ratio—2.88 to 1.
3. Draw range—±4.5% from mean output speed.
4. Horsepower—A.G.M.A. ratings of 150, 300, 600, 900, and 1200.
5. Maximum acceleration horsepower—twice A.G.M.A. ratings.
6. Speed regulation—0.007% at full load.

Testing

In order to determine the performance of these drives, it was necessary to subject them to exhaustive tests under laboratory conditions, as it would be impossible to make the necessarily accurate measurements in the field (see Fig. 6.) Two prototype drives were built, in order to buck one drive against the other. The system used to load the drives is analogous to a gear tester (see Fig. 7). With this method, large powers are circulated, in a circle, with only losses and the speed being regulated by the lineshaft motor. By varying the voltage on the booster generator, the total

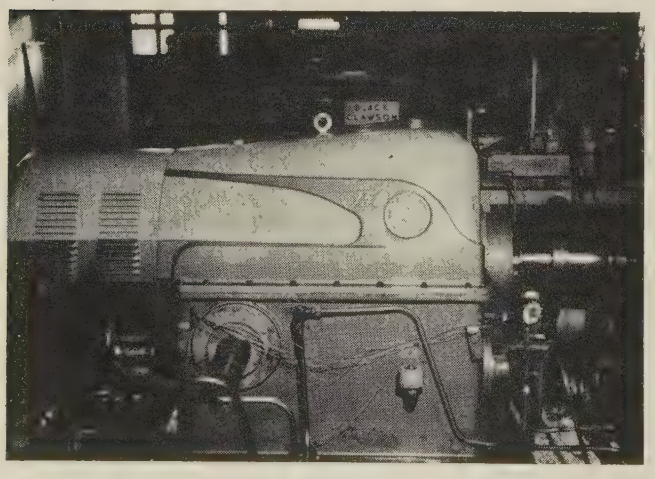

Fig. 9

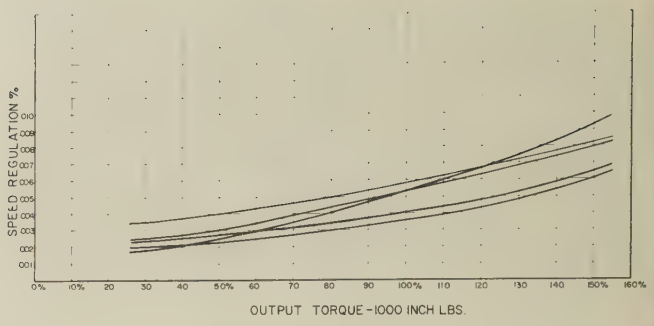

Fig. 10. Differential drive unit speed regulation

load, through the drives, could be adjusted to any desired value. Also, by disconnecting the loading generator, the load could be dropped instantaneously.

In order to detect the effect of load variations, it was necessary to use a stroboscope arrangement as shown in Fig. 8. The output shaft of each drive was connected to a jack shaft, with a five to one speed-up ratio. From one of the jack shafts, a signal was taken which flashed a strobe light upon a marked disk on the other jack shaft. The combined effect of the speed-up plus opposite reactions of the two drives gave an error magnification of ten.

It was also necessary to take temperature readings throughout the drive in order to check on the lubrication efficiency. Thermocouples were inserted through the drive case and attached to various bearings (see Fig. 9.) By this method, several changes to the oiling system were discovered to be necessary and when the changes were made, the temperatures dropped to normal values.

Regulation

The results of the regulation tests are as shown in Fig. 10. In this graph, the power is expressed as per cent load with 100% being equal to the A.G.M.A. rating of the gears. It can be seen that the per cent regulation increases as the load increases with very little change for the different draw rates. At 100% load the regulation is 0.005%. These tests were run at various lineshaft speeds and in all cases were nearly identical.

It was also desired to know the speed regulation of constant load and speed over a prolonged period of time. The drives were set up for maximum speed and rated load and were checked over a period of 200 hr. (see Fig. 11). It was found that the regulation, under these conditions, was ±0.0021%. When this value is added to the variable load regulation, the total variation is 0.007%.

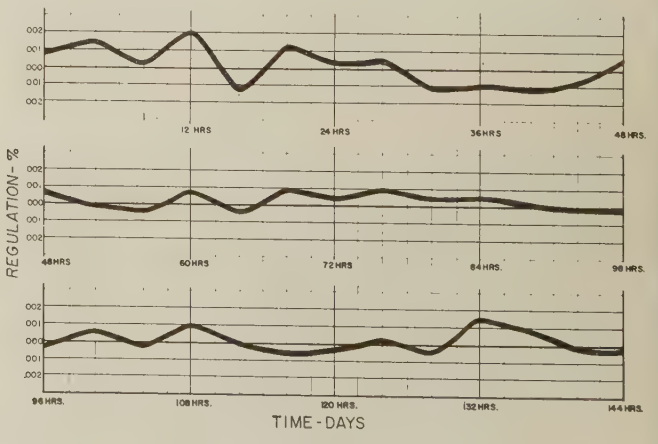

Fig. 11. Differential drive unit

Speed regulation drift over a 144-hr. time period. Load, 155 hp.; speed, 1000 r.p.m.; draw, +3.2%.

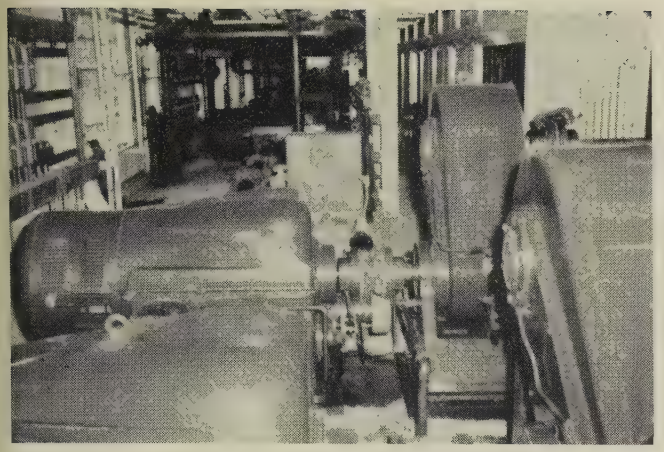

Fig. 12

Application

The actual installation of a differential drive requires that an additional gear train be installed between the differential and the in-drive shaft, in order to establish the proper speeds. These gear trains are simple parallel shaft reducers and are made in many ratios in order to meet any machine conditions. Various lengths of transfer cases are available to provide the proper in-drive locations. The following photographs show an actual installation of the drive (see Figs. 12 and 13).

SELECTION OF DRIVES

After discussing the different types of drives available, the question then arises as to which is the proper drive for a specific installation. There are several factors to be considered in this question, such as machine size and speed, type of products to be made, cost and the degree of maintenance which must be provided. Experience with the various factors involved is invaluable when making this decision.

Horsepower

If we assume that a mechanical drive is to be applied to a fourdrinier and the normal application factors for this type of drive are used, the maximum horsepowers available from the various types is as shown by Fig. 14. Flat belt drives can be used up to 340 hp., V-belt drives up to 400 hp. and differential drives up to 800 hp. All of these horsepower values are based upon actual running loads.

This information can be shown in another form (see Fig. 15). Assuming that the normal running load factor is 0.086 for a fourdrinier, we can plot the width of wire versus the machine speed for the maximum horsepower available from the various type drives. If the required horsepower is above the 400 hp. range, there is no question as to the type of drive,

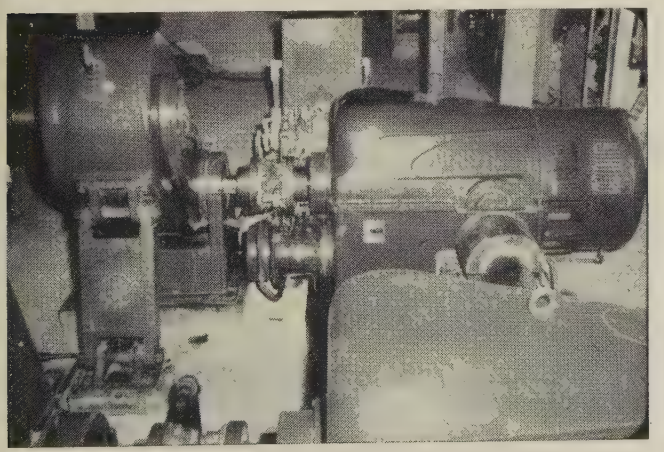

Fig. 13

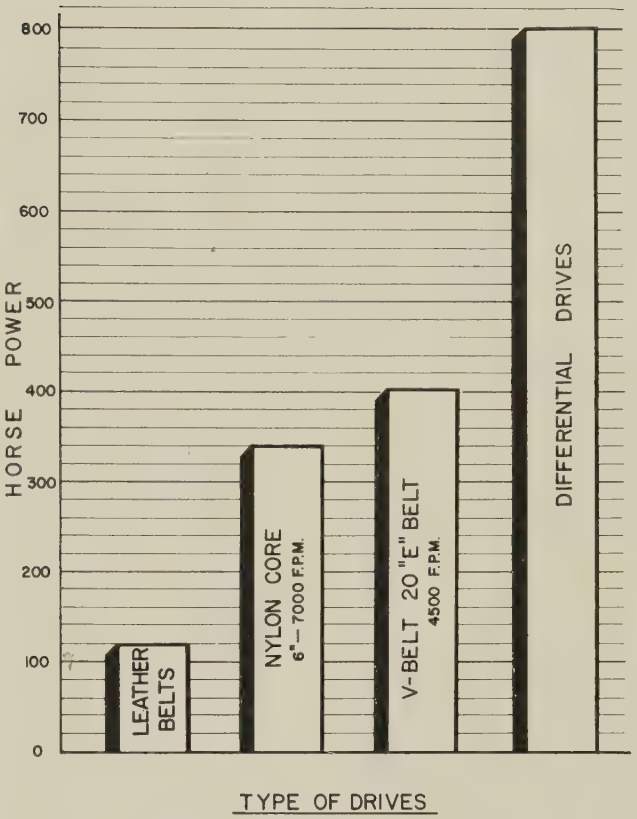

Fig. 14. Horsepower comparison of available mechanical drives

as only a differential drive is large enough. If we are below this range, we must use some other additional requirements to aid in the decision.

Regulation

The degree of regulation which is required is dependent upon the machine speed and the type of product. As machine speeds increase, minute variations in draw become more violent, with the result that there is a great deal more chance of breaking down the sheet particularly at the wet end.

The type of product also affects the regulation requirements. The weaker or more tender the sheet, the closer the regulation must be held. For instance, a brash, tender sheet made from semichemical pulp must be handled much more gently than a heavy kraft sheet. A tissue must be handled differently from a board.

If rigid regulation is not required, the flat belt drive will meet the requirements very satisfactorily. Where a more closely controlled regulation is required, the V-belt drive is

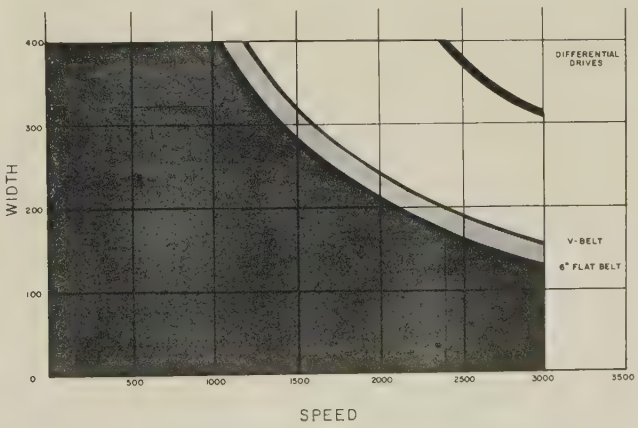

Fig. 15. Range of application of mechanical drives

indicated. If you require the ultimate in accuracy, the differential drive meet the requirements.

Under certain conditions, the use of a more rigid regulation, than that required to produce the desired result, can lead to problems further along the machine. This is particularly true where the sheet has very little shrinkage or stretchability, during the drying process. Under these circumstances, the drive must have some flexibility in order to allow the various draws to vary slightly, when the demand arises, so that sagging or breaking of the sheet does not occur. Therefore, where certain accuracies are required for the proper operation of the wet end of the machine, it may be necessary to deliberately destroy that inherent accuracy in the dry end of the machine.

Cost and Maintenance

The least expensive, and easiest to maintain, is the flat belt drive. Slightly higher in cost and a little more difficult to maintain is the V-belt drive. The differential drive is the most costly, about equivalent to a sectional electric drive, and it requires not only a knowledge of mechanical drives but also a knowledge of hydraulics.

CONCLUSION

Since the mechanical type drives have now been expanded to meet the requirements of all sizes, speeds, and types of paper machines, we now have a choice of drives available. It is with this choice, that we can be of help, in order to insure that you get the drive most suitable for your individual requirements. To do this requires that consideration be given to all of the previously mentioned points and each must be weighed against the others. If this is done, the drive selected will be tailor-made to suit your application.

Presented at the Thirteenth Engineering Conference of the Technical Association of the Pulp and Paper Industry, Portland, Ore., July 28–Aug. 1, 1958.

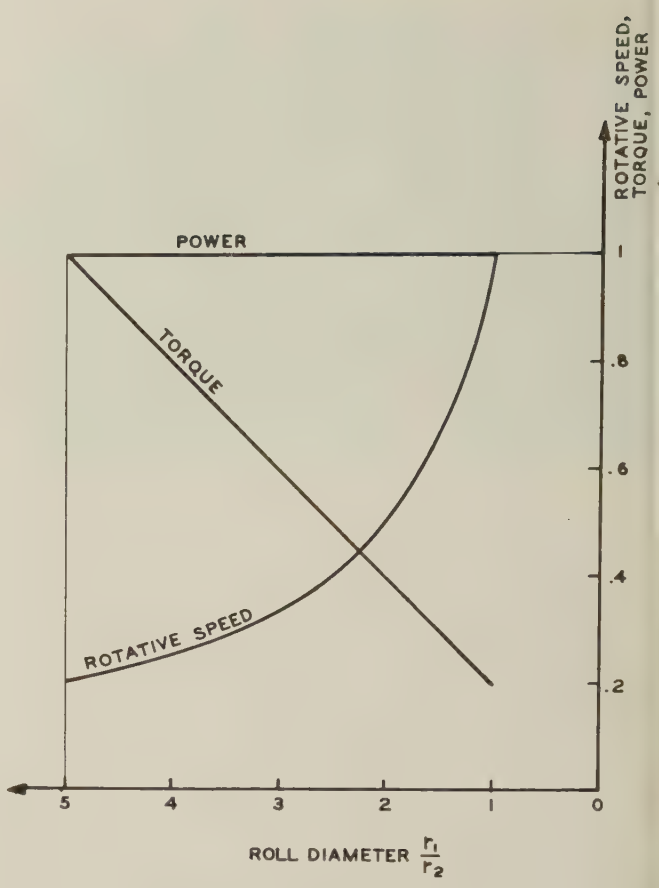

Fig. 1. Rotative speed, torque and power versus roll diameter

Power Demand Decrease and Recovered Energy by Regenerative Braking of the Unwound Paper Roll

HENRY SCHREIER

In the selection of a braking method to be applied to the unwound paper roll in a winder, supercalender, or any other machine requiring unwinding, important factors are the decrease of the power demand and the value of electric energy that can be regained when applying regenerative braking in contrast to frictional braking in which energy is turned into heat and dissipated. In the following a short-cut way of arriving at these data shall be presented.

As first, the energy that would be lost by frictional braking shall be determined, with the limitation to cases where automatic regulation of the tension in the sheet is being applied to obtain the required quality of the rewinding.

With this the tension in the sheet can be assumed constant during the entire rewinding of a roll, and the exerted force is:

$$F = w\,f = \text{const.}$$

where

w = the width of the sheet in inches,
f = the tension in the sheet in lb. per inch of width

The braking torque applied to the roll at any instant is:

$$M = Fr$$

where

r = the momentary value of the roll radius

Henry Schreier, Staff Engineer, Beloit Iron Works, Beloit, Wisc.

This radius decreases in course of the unwinding from its value r_1, which is the radius of the full machine or parent roll, to r_2, the radius of the core.

The power dissipated as heat in a nonregenerative brake, when neglecting the power lost in the bearings as being small in comparison to the former, is expressed by the formula:

$$P = \frac{Fv}{60} \text{ in foot pounds per second}$$

or

$$P = 0.226\,Fv \times 10^{-4} \text{ kw.}$$

where v denotes, in feet per minute, the speed of the paper as it comes from the unwound roll. This speed v is constant. With F = constant, as shown before, it follows that also P = constant. For the sake of completeness also the formulae or the rotational speed of a roll at any moment is given:

$$n = \frac{v}{2r\pi} = \frac{\text{constant}}{r}$$

which is the equation of a hyperbola. The above derived equations are shown graphically in Fig. 1. The energy, in kilowatt-hours, dissipated in the brake during the unwinding of a roll is expressed by:

$$E = \frac{Pt}{60} \text{ in kw.-hr.}$$

where t is the time of unwinding a roll in minutes. This time can be calculated from:

$$t = \frac{(r_1 + r_2)2\pi}{2} \times \frac{r_1 - r_2}{c} \times \frac{1}{12v} = \frac{(r_1 - r_2)^2\pi}{12cv}$$

where c denotes the thickness, or caliper, of the paper in inches. By substituting for t in the formula for E one arrives at:

$$E = \frac{0.226\ wfv}{60} \times \frac{\pi(r_1 - r_2)^2}{12cv} \times 10^{-4}$$

$$= 0.0314 \frac{f}{c}\ w\pi(r_1 - r_2)^2 \times 10^{-6}$$

$$= 0.0314 \frac{f}{c}\ V\ 10^{-6} = 0.0314 \frac{f}{c} \times \frac{W}{d} \times 10^{-6}$$

where

V = the volume of the roll in cubic inches
W = the weight of the roll in pounds
d = the density of the paper in pounds per cubic inch

For easy calculations of economics of regenerative braking the denomination e "specific energy losses" is introduced. It denotes, in kilowatt-hours, the average quantity of energy dissipated in a mechanical brake per pound of paper of an unwound roll.

$$e = \frac{E}{W} = 0.0314 \frac{f}{cd} \times 10^{-6}$$

The somewhat unusual magnitude c, the caliper of the paper, can be substituted by another one that can be easily derived from the ream weight:

$$W_R = N(ab)cd$$

where it denotes:

N = number of sheets in the ream
(ab) = the size of the sheet in sq. in.
$cd = \frac{W_R}{N(ab)} = s$
s = the weight of the paper per square inch

Finally one arrives at:

$$e = 0.0314 \frac{f}{s} \times 10^{-6} \text{ in kw.-hr./lb.}$$

or

$$e = 0.0628 \frac{f}{s} \times 10^{-3} \text{ in kw.-hr./ton}$$

The further course of calculations is as follows: the yearly manufacturing schedule, known or anticipated, specifies the quantities per year and ream weights of the various grades of paper that will require rewinding. Using the formula for e, the specific energy rewinding losses per ton can be calculated for each grade of paper included in said schedule. Where the actual values of the applicable sheet tensions for said grades of paper are not available, approximate values can be derived from the curve given on TAPPI Data Sheet 145 D or from those shown on Fig. 2. By multiplying the so-determined specific losses by the respective yearly quantities of paper one arrives at the quantity of energy that would be lost per year in rewinding with a friction brake. Even with regenerative braking not all of that energy can be recovered though, as the unwind stand and the machines contained in the setup for regenerative braking have their unavoidable losses. These losses are taken care of by introducing an efficiency factor, and the formula for the recoverable quantity of energy is

$$E_R = e\ Q\ \eta$$

where Q = yearly quantity of a certain grade of paper.
A practical value of the efficiency factor is $\eta = 0.7$. An example may further elucidate the matter. The energy shall be determined that can be recuperated by regenerative braking on a winder on which 90,000 tons per year of kraft bag and wrapping paper are to be rewound. Of these

67,500 tons are of 40 lb. per ream substance
14,400 tons are of 60 lb. per ream substance
8100 tons are of 80 lb. per ream substance

For the 40-lb. per ream paper

f = 2.5 lb. per in.
$s = \frac{40}{500\ (24 \times 36)} = 0.0926 \times 10^{-3}$ lb./cu. in.
$e = 0.0628 \frac{2.5}{0.0926} = 1.696$ kw.-hr./ton
$E_R = 1.696 \times 67,\ 500 \times 0.7 = 80,200$ kw.-hr./year

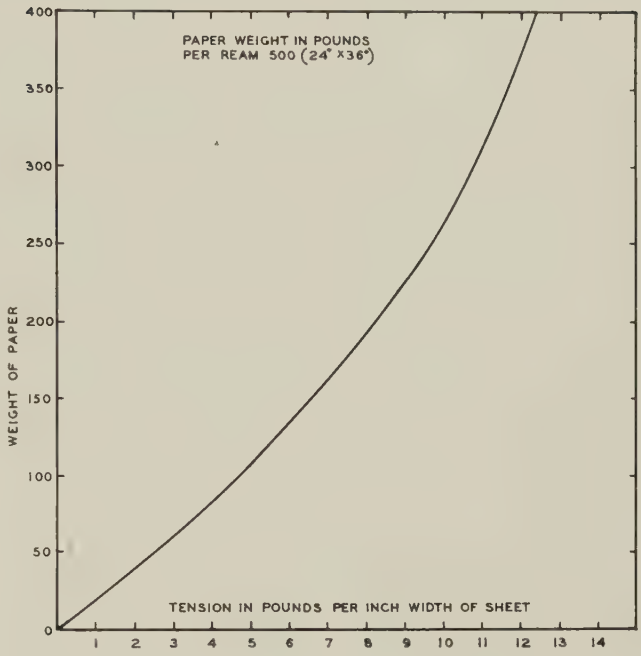

Fig. 2. Winding tension versus paper weight (after L. W. Fisher)

For the 60-lb. per ream paper

f = 3 lb. per in.
$s = 0.1389 \times 10^{-3}$ lb./cu. in.
$e = 0.0628 \frac{3}{0.1389} = 1.357$ kw.-hr./ton
$E_R = 1.357 \times 14,400 \times 0.7 = 13,690$ kw.-hr./year

For the 80-lb. per ream paper

f = 4 lb. per in.
$s = 0.1852 \times 10^{-3}$ lb./cu. in.
$e = 0.0628 \frac{4}{0.1852} = 1.355$ kw.-hr./ton
$E_R = 1.355 \times 8100 \times 0.7 = 7690$ kw.-hr./year

The total quantity of energy recoverable by regenerative braking is consequently 101,580 kw.-hr. per year.

When figuring the money value of the recovered energy in the case of a mill generating power from steam going to a condenser and/or purchasing energy from outside, the cost of that kind of energy should be used the quantity of which would be reduced by recuperation.

The power demand of a drive which includes a braking generator is smaller than of one with a frictional brake, as in the former only the losses have to be met by the electric system. Thus, seemingly, the generating capacity of the power plant could be made smaller by a corresponding amount, or in an existing one a part of it released and made available for another use. These savings though are only seldom taken into consideration, because they appear to be practically unattainable. The decrease of the power demand, even when applying regenerative braking on all unwind stands, is so small when compared with the power plant capacity that it is mostly of no consequence. Sometimes though such an approach is not justifiable. Besides the regenerative braking there may be also other measures that would influence the power demand of the mill in the same manner. In certain cases these measures, when considered as a complex, instead of each separately, may have a bearing on the power plant capacity and, consequently, lead to economies that deserve not to be overlooked.

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Plastic Reinforced Flat Belts

RICHARD H. RHOADS

Flat belting has made an important contribution in the field of industrial power transmission ever since the start of the industrial revolution. A flat belt running over two or more pulleys is simple to design, install, and maintain. This type of drive is adaptable to a wide range of loads, speeds, and driving conditions, and when properly designed and installed has given extraordinarily efficient and economical operation. Long center flat-belt drives have run for 20 or more years with little attention. There has, however, been a strong tendency to turn to other types of drives, some of which are far more complicated and expensive.

Perhaps the biggest single difficulty which industry has experienced with flat belts has been inability to maintain the desired tension. In addition, flat leather belts have not run as true on the pulleys as industry wants. In other words, flat belts stretched and wobbled. Some of the leading flat belt manufacturers have undertaken extensive research programs to try to solve this problem.

To perform most satisfactorily belting should have a small amount of elasticity but no permanent stretch. Numerous unsuccessful efforts have been made to reinforce belts with steel bands or other relatively inelastic materials.

Moderate elasticity cushions shocks caused by sudden variations in load or minor mechanical inaccuracies, such as slight pulley eccentricity or shaft wobble. This elasticity also prevents slight inaccuracy in belt length at installation, or changes in belt length produced by changes in temperature or moisture, from causing critical changes in belt tension. There are a number of belts on the market which can quite readily take care of the drives of no more than average difficulty, but the problem, especially in the paper industry, has been to find a belt which can perform well under the increasingly severe conditions of heavier loads, higher speeds, and requirements for a more even draw.

It was a little over 2 years ago that the first plastic reinforced flat belts appeared on the American market and for the first time a belt was available which, in a large measure, combined the best features of a top quality flat leather belt with a very substantial reduction in the amount of permanent elongation throughout its life. In fact, this reduction in stretch has been sufficient so that it is very rare for a well designed plastic reinforced belt to require any adjustment after it has been installed.

Results obtained in service on hundreds of drives confirm observations from a simulated life testing procedure which was developed in our laboratory to measure belt elongation under controlled conditions. Four weeks' exposure to the test procedure that will be described, gives results approximating a year or more under normal operating conditions.

In this test a 1-in. wide belt 5 ft. long, made endless, is placed over two pulleys mounted one above the other. The upper pulley is 6 in. diam. and the lower pulley 12 in. diam. is made of solid cast iron, and mounted on an arm so that a 60-lb. weight maintains tension in the belt.

The test begins after the belts have been kept in a conditioned cabinet for 1 week and four 10-in. base lengths laid out on the back of the belt when it is not under tension. The belt is now exposed to four steps of increasing severity and the elongation measured periodically. During the first few days the belt is static except that the pulleys are turned occasionally by hand to permit the belt over the pulleys to elongate. During the second week the belt is transferred to similar pulleys but the upper pulley is run at 1000 r.p.m. The flexing of the belt usually induces additional stretch.

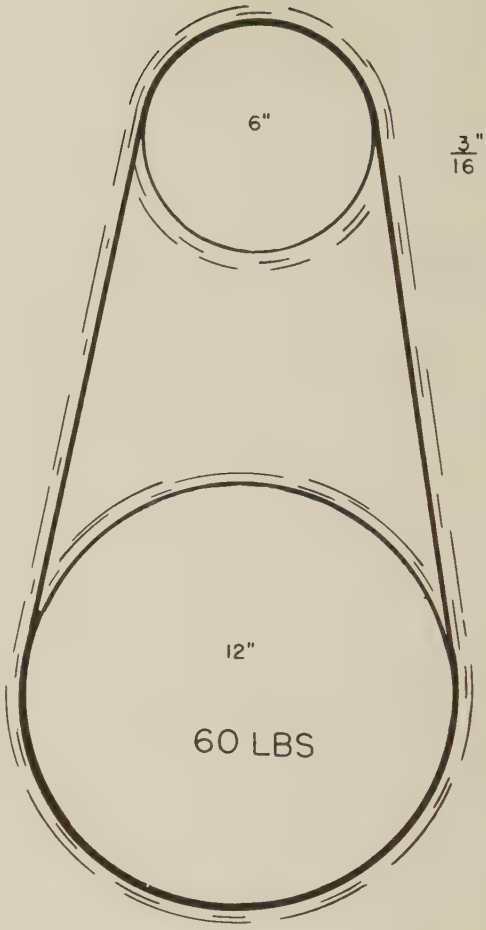

Fig. 1. Eccentric pulleys

When a load is transmitted by a belt one strand of the belt is tight and the other relatively slack and consequently a given segment of the belt is alternately tightened and loosened as it goes around the pulleys. This condition is duplicated in the third week of the test by running the belt on similar pulleys but the upper pulley is mounted with $^3/_{32}$-in. radial eccentricity. It has been found that this eccentricity induces tensions in the belt that approximate the tensions caused by operation at rated loads.

During the final week the belt runs on similar pulleys except that the upper pulley has $^3/_{16}$-in. radial eccentricity. This approximates severe overload conditions. Figure 1 shows the general arrangement of pulleys and Fig. 2 is a chart showing the per cent elongation of a typical oak tanned belt.

From practical experience we have learned that if a leather belt elongates 8% of its initial free length, it will probably continue to elongate to rupture and that it is wise to take it off while it can be used elsewhere and install a belt with greater capacity.

Figure 3 shows the results from a cord reinforced rubber belt. The initial elongation under static tension is low but remember that because rubber has a lower coefficient of friction than leather, rubber belts need to be run at higher tensions to carry the same load as the oak-tanned leather. Also notice that the test could not be carried through on the high eccentric drive. When this was attempted the cords in the belt were not sufficiently elastic to absorb this much shock and the lower pulley bounced and pounded so that the test could not be continued.

Richard H. Rhoads, J. E. Rhoads & Sons, Wilmington, Del.

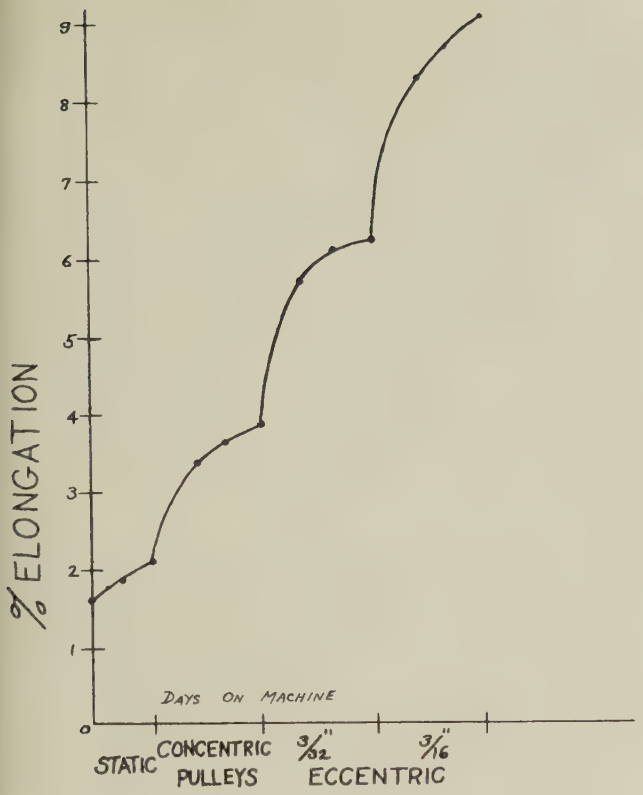

Fig. 2. Oak leather

Our laboratory has made numerous attempts to find a reinforcing material to reduce the stretch and Fig. 4 shows the typical result of a leather belt which has been reinforced with Dacron cord. Notice that the elongation and type of curve is very similar to that of the rubber belt and again it is not capable of withstanding the severe shock loads of running over the pulleys which are $^3/_{16}$-in. eccentric.

Figure 5 illustrates the elongation in the Tanastic belt which is one of the plastic reinforced flat belts. Notice that for the first time we have a belt which has a very low amount of elongation even under heavy loads and which is also capable of holding up on the $^3/_{16}$-in. eccentric pulleys. Prompt acceptance of this type belt on paper mill cone drives is an indication not only of the need for such a belt, but of the alertness of the paper mill engineers in quickly recognizing a product that met their needs.

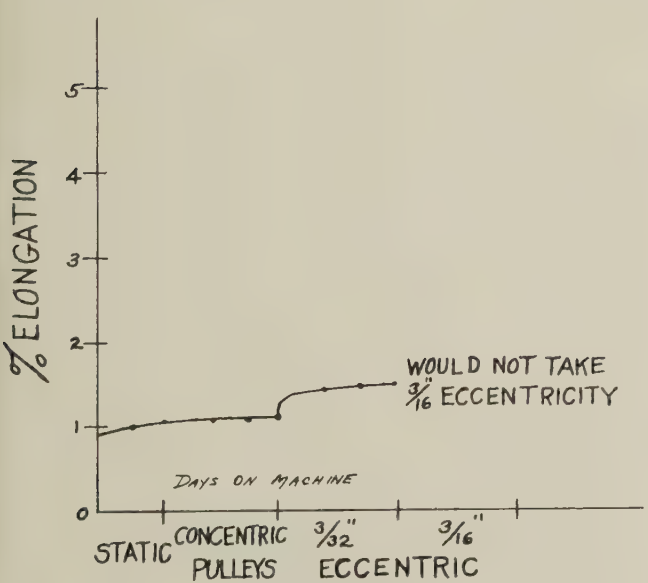

Fig. 3. Rubber belts

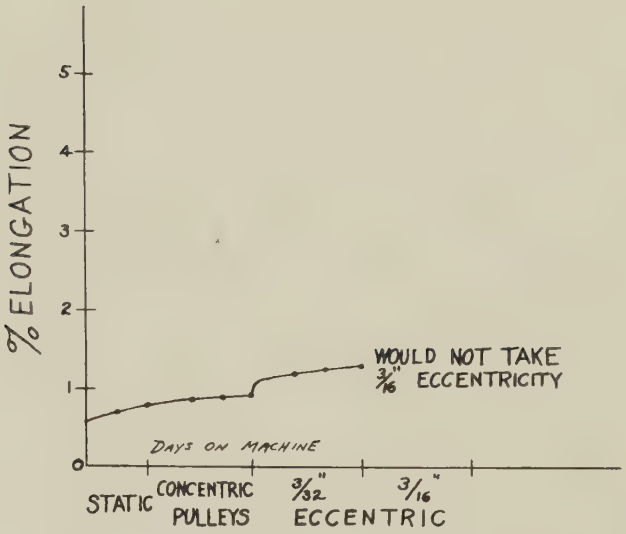

Fig. 4. Tannate leather reinforced with Dacron

Elongation, of course, consists both of elasticity and of stretch and it's interesting that in the Tanastic type of belt the elongation which we have is almost entirely elasticity and at the conclusion of these tests, when the belt has been removed, it will return nearly to its original length.

Figure 6 shows the oak leather and plastic-reinforced belt plotted together for comparison. Each of these is a typical curve and represents the results of scores of tests and in some cases of several years of testing.

Nylon core belts weigh less than conventional belts of the same capacity. This reduces the centrifugal effect and permits operation of belts at higher speeds than is practical with either straight leather or rubber. Six to seven thousand feet a minute is the practical limit for leather and rubber belts, but 10,000 or 12,000 f.p.m. is entirely practical for the weight nylon core belts usually used on paper mill cone drives. Ductile iron pulleys are now obtainable that are safe in these speed ranges and this combination of belt and pulleys lifts a limit which was being felt in the design of paper machines to a point where belt speed is no longer the limiting factor. In addition, a belt which is elastic and which does not have permanent stretch can operate over very short centers. A belt has been run over cone pulleys on 27-in. centers in a paper mill which was still running after more than 2 years without any adjustment or attention.

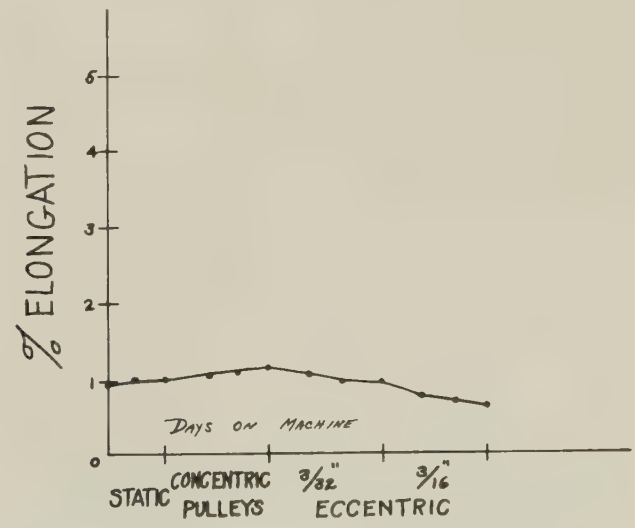

Fig. 5. Tanastic

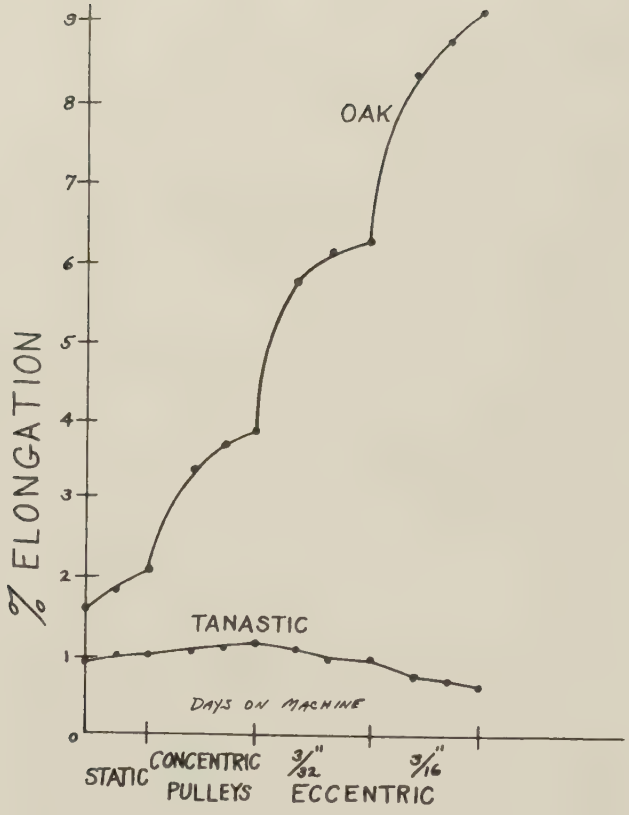

Fig. 6. Comparison oak—tanastic

The second problem which was mentioned at the start of this discussion was that of a belt which would run more true, especially on cone pulleys. When a flat belt runs over cone pulleys the speed of the driven pulley is, of course, affected by the relative position of the belt on the cones. The effective line of pull which determines the driven speed is usually about in the center of the belt.

All belts running over cone pulleys are by the nature of the drive forced to slip some on the outer edges. Within limits this slip is not significant in terms of performance providing the effective line of pull stays in one place on the belt. The wider the belt and the less uniform in quality it is throughout its width, the more likely it is that this effective line will wander and as it wanders, the speed of the driven pulley tends to fluctuate even if the belt remains in the same place on the cones. The new plastic-type belt runs true on the pulleys and since the oriented nylon is not only more uniform throughout its width but also is usually narrower, the effective line of pull stays within a very narrow range and consequently there is a more uniform draw and less pressure against the shifters. In other words, the second major problem has also been solved and so far as we know to the satisfaction of the industry.

Nylon-core belts are made with a leather surface on both sides, or on the pulley side only with fabric on the other, or with one or both sides covered with synthetic rubber. The bare nylon core is not suitable for a belt because its low coefficient of friction does not give satisfactory grip on the pulleys. Leather on both sides permits using the belt on both sides, either when the drive is so arranged or so that the belt can be turned over if one surface becomes worn or damaged. Leather on both sides also presents more wearing surface to a shifter.

The belt with leather on one side and fabric on the other usually sells for 15% less than the belt with leather on both sides. It is slightly lighter and, therefore, less affected by centrifugal tension, but has no other significant advantages.

More than one variety of nylon is being used in belting offered in the United States. Some of these are less elastic and have more tendency toward brittleness and longitudinal fracture than others which are more elastic and more pliable. A proper balance between these characteristics is important. It is also well to know that nylon has a tendency to become slightly less pliable as it ages.

Nylon-reinforced belts are made with various thicknesses of nylon core, varying from less than 24 to 216 thousandths of an inch, the thicker cores being made of laminations of thinner ribbons. The ultimate tensile strength of the core alone under normal atmospheric conditions is in the range of 20,000 to 30,000 p.s.i. and the elongation at rupture about 50%. Ultimate tensile strength increases when the plastic is very dry and is lower when very wet. Belts are normally designed with a factor of safety of approximately 10 to 1. On this basis, plastic belts are frequently one half to one third the width of conventional leather or rubber belts for the same drives. Narrower belts are particularly helpful on cone drives as this reduces the forced stretch at the edges of the belt.

The narrow width of nylon core belts carry the same tensions as the wider widths of conventional belts. Their lesser weight per linear foot often gives the impression that the belts are excessively tight, just as a wire carrying the same tension as a rope seems tighter than the rope.

Tensions in nylon core belt are determined by making the belts a designed percentage shorter than the tape line length around the pulleys. Initial length deductions of 2 to 4% depending on conditions is the usual range of allowance to produce initial belt tension.

These plastic reinforced leather belts do have one important disadvantage. More skill is required to prepare the laps than is required with leather belting. However, any good mechanic can be trained to do this work and some tools have been developed in the last year which make the work easier. Cementing the joint of a leather-faced nylon-cored belt takes more pressure and a longer time than for an all leather belt and 8 hr. in the press is recommended. A new line of all plastic belts is now available which has the same nylon core as the Tanastic-type belt but with a fabric and synthetic rubber surface. The laps on this new all-synthetic belt can easily be beveled by machine and without skilled labor in a matter of a few minutes. Even more important, the laps on this all-synthetic belt can be cemented in a heated press and the cement will dry in 35 to 40 min. on the heavy weight belt and an even shorter time is required for the lighter weight belts. The surface on these all-synthetic belts is not so rugged as that on the belts with the leather surface. A number of these belts have now been run on paper machines for 6 months and while they are not recommended for the larger horsepower drives, they do seem to have a real place in the pulp and paper industry.

These belts with synthetic rubber on one or both sides have a lower coefficient of friction and consequently a lower capacity size for size than a belt with a leather surface. They are less expensive than the belt with leather and are slightly lighter and more flexible.

Some paper machine operators are further reducing the possibility of lost production through unscheduled shut downs by carrying spare belts made endless around the shafts of cone pulleys which can be run on the cones in a few minutes. This plan of running belts onto pulleys is also used instead of drawing the belts tight with belt rods and heads. It becomes more difficult with wider belts but devices have been developed, which will not be described here, that make this safer and easier than may seem apparent.

The nylon core can be operated in a temperature range from −40°F. to +160°F. It tends to shrink slightly at higher temperatures and at lower humidities. Since high temperatures are usually accompanied by high humidity, the net effect is not enough to create a problem except under

extreme conditions. It is recommended practice to store a belt near the machine on which it will be used, but many mills not doing this are not experiencing difficulty. This difference in length from atmospheric conditions may show up as a long gentle curve in a belt rolled out on the floor, if the belt has been stored in a roll with one edge up and the other down.

As mentioned earlier, all the nylon core belts tend to become slightly stiffer with age and this tendency is accelerated at elevated temperature.

Belts of this type have been used in Europe since the late 40's. Generally, the paper machines there are smaller and slower speed than the latest machines here. The author visited quite a number of these mills and found users enthusiastic about the improved results obtained with these belts. They advise that the normal failure of a belt of this type, when it wears out, is a failure in the plastic. This is most likely to occur at or near the endless lap. We have seen a few failures caused by separation of the leather from the plastic or separation between the plies of plastic and some caused by poor handling in making the endless lap, but the great majority of the belts installed in this country in the last two years are still operating and giving exceptionally good service.

Presented at the Thirteenth Engineering Conference of the Technical Association of the Pulp and Paper Industry, Portland, Ore., July 28–Aug. 1, 1958.

Paper Mill Roll Grinding by the Abrasive Belt Method

JOHN BURROUGHS

In the field of roll grinding and finishing, grinding wheels have been and are the standard medium while coated abrasives have, for a long time, been used only for a few finishing operations. In the last two years, a new proved concept in the grinding and finishing of all types of paper mill rolls with coated abrasive belts has been achieved. Recent developments have made it possible to rough grind and finish rolls by this method in substantially less time while maintaining the rigid tolerances required. Proper application of this method is producing great savings in labor and overhead on the roll grinding operation while reducing production down time.

Paper mill rolls must be concentric, with little or no taper, and be free of chatter, traverse or spiral marks, fish tails, or any other undesirable effects. Therefore, the selection of the proper contact wheel and abrasive belt has become as important as the specification of the grade, structure, and bond for a grinding wheel application. The contact wheel should be selected only after careful consideration has been given to all conditions present. This includes machine condition, material to be ground, stock removal required, degree of accuracy desired and undesirable outside influences. The proper type of abrasive belts and belt splices can be determined only through the type of contact wheel selected and the type of material to be ground, including chilled iron, steel, brass, rubber, etc.

A few case histories may be of interest. One of these involved the roll grinding department of a large manufacturer of precision roll grinding machines where the writer conducted tests during the week of Oct. 27, 1958.

The roll to be ground was a chilled iron roll 18 by 274 in. Diameter after grinding was within 0.0005 from end to end and concentricity was in the low tenths. The microinch RMS reading was 4 and a decided absence of fish tails was noted, the latter proving to be characteristic of the abrasive belt method. Time required to produce these results was less than 50% of the method formerly used, and using *only one side* of the grinder! Normally, two sides were used when employing the grinding wheel method.

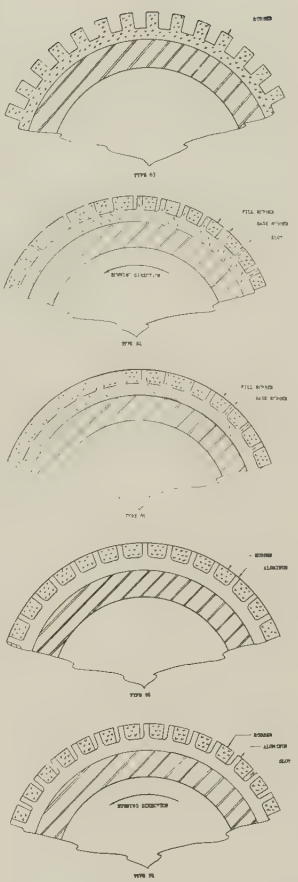

Fig. 1.

In a large southern pulp mill we ground a king roll, 32 by 205 in. The material was chilled iron of 53 Rockwell C hardness with many burned spots and deep pits. The head-stock end of the roll was 0.005 in. larger than the tailstock end and the crown measured 0.038 in. Requirements were to crown the roll between 0.045 and 0.050 in. and maintain a taper of less than 0.001 in. Roll measurements before grinding were 31.911, 31.949, and 31.916 in. The first reading after grinding was 31.907, 31.945, and 31.907 in. The crown was not improved, but the ends of the roll were brought within tolerance. Next measurement was 31.893, 31.939, and 31.893 in. This brought the crown and taper within tolerance, but deep pits and burned spots were still in evidence. It remained to remove these undesirable conditions and still maintain the required tolerances. After continued grinding, this was accomplished. When the roll was finished, it was inspected for diameter, taper, crown, and surface condition. The roll was accepted with readings of 31.880, 31.928, and 31.880 in. Taper from end to end did not show any difference in the straddle gage and crown was 0.048 from both ends. Usual grinding time with two bonded wheels was 50 hr. and the abrasive belt time, using only one side of the machine, was 25 hr.

In another southern kraft mill we were required to rough grind and finish a chilled iron queen roll, 24 by 264 in. This roll could be crowned 0.003 and a tolerance of 0.0015 was allowed from end to end. In this particular case, when the operator was finishing the roll by the bonded wheel method and the paper machine winder was in operation, he had to remove the wheel from the roll. Vibration set up by the winder was being transmitted through the floor to the grinder and the grinding wheel, resulting in marking the roll. When the abrasive belt method was tried, the contact wheel over which the abrasive belt runs was resilient enough to absorb the vibration and allow the operator to finish the roll without stopping. This roll required 36 hr. by the bonded wheel method, but through the use of the coated abrasive belt and contact wheel, the time was reduced to $6^1/_2$ hr. When finished, roll readings were 23.8165, 23.8195, and 23.8175. There is no question in the author's mind that the roll could have been brought into more exact readings, but the company considered the roll well within tolerances to perform the task for which it was intended.

Another paper mill requested that we regrind their rubber rolls to ascertain the efficiency of the abrasive belt method. The first roll was a rubber size press roll 31 by 240 in. made of

John Burroughs, Coated Abrasives Division, The Carborundum Co., Niagara Falls, N. Y.

80 durometer rubber and requiring a crown of 0.060. This roll required 8 hr. to finish by their bonded wheel method. After $1^1/_2$ hr. the roll was finished and readings were: 0, 10, 19, 28, 37, 44, 50, 54, 57, 59, 60, 59, 57, 54, 50, 44, 37, 28, 19, 10, 0. Before grinding readings were: 4, 0, 14, 26, 40, 50,54, 60, 65, 66, 59, 62, 62, 60, 60, 53, 46, 70, 17, −8, 0. The second roll was a top wet press roll 31 by 240 in., also of 80 durometer rubber and requiring a crown of 0.150. The operator normally took 8 hr. to grind this roll, but floor to floor time by the abrasive belt method was 1 hr. and 50 min.

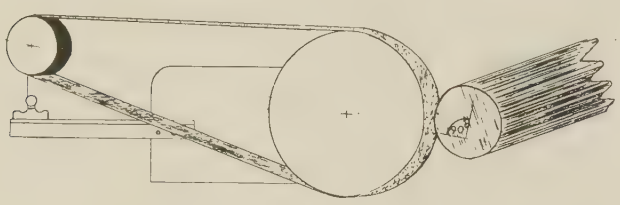

Fig. 2.

Readings after grinding were: 0, 23, 47, 70, 91, 108, 123, 135, 144, 148, 150, 148, 144, 135, 123, 108, 90, 70, 46, 23, 0. Readings before grinding were: 0, −25, 59, 82, 100, 120, 130, 140, 157, 165, 169, 169, 166, 154, 153, 143, 121, 99, 77, 50, 0.

The preceding actual case histories are excellent examples of the efficiency obtainable by converting to the abrasive belt method. The belt method requires only modest outlays for additional equipment and is becoming widely-accepted throughout the United States and Canada.

MACHINE CONDITION IMPORTANT

Roll grinding with abrasive belts requires machine rigidity and good over-all condition of the machine and its attachments, much as these conditions are vital to the proper and effective use of grinding wheels. Accuracy obtainable by the belt method is equal to the accuracy of the machine itself. When roll grinders are converted to belt usage, the belt will produce whatever accuracy the wheel produced and do it, in most cases, in substantially less time.

It has also been proved that the abrasive belt-contact wheel method will permit effective roll grinding and finishing more readily when machine conditions leave something to be desired for proper results with the grinding wheel method. This is due mainly to the following reasons:

1. Contact wheels may be made in varying degrees of hardness.
2. Contact wheels can be made to produce any degree of aggressiveness desired.
3. Contact wheels have a radial depth of face covering on the wheel which governs to a large degree the resiliency which can be obtained from a wheel of a given hardness.
4. Contact wheels can be serrated which determines rate of cut and uniformity of finish produced by a belt during its useful life.
5. Contact wheels can be made of rubber, dual durometer rubber, or metal and rubber.

CONTACT WHEEL SELECTION

With the aforementioned reasons in mind, it will be obvious that different types of contact wheels are recommended for different machine and work conditions. The same abrasive belt will act differently when used with various types of contact wheels, each of which serves a purpose which the others cannot achieve. For very good to excellent machine conditions, one of the several choices in the "90" series is recommended. For fair to good machine conditions, one of the several choices in the "80" series is suggested. With anything less than fair machine conditions, tolerances produced on rolls cannot be maintained closely, and a choice in the "60"-series is recommended. Both the "80" and "90" series contact wheels are covered by Carborundum's U. S. pat. 2,865,-144.

It is most interesting to note why different types of contact wheels make a belt act so distinctively. The "90" series contact wheels, when used properly, will eliminate any out-of-round, taper, or surface imperfections more quickly and effectively than any other type of contact wheel known to the trade today. Further, they will give satisfactory finishes on many types of rolls in most cases. While the "60" series contact wheels are far from being the choice for producing close tolerances, they will give the best finishes.

The "80" series contact wheels, made of dual durometer rubber, will produce very close tolerances and still be resilient enough to absorb vibration and produce a good finish. Heavy pressures cause it to hog off stock while light infeed pressures permit the belt to produce a good finish.

Some of the available types of contact wheels are shown in Fig. 1.

CONTACT WHEEL BALANCE

Most contact wheels are in good balance when received from the manufacturer. However, balancing of the contact wheel, at operating speed, is an absolute must regardless of the perfection of the roll grinder; since any out-of-balance condition is greatly magnified.

The Curtis Machine Div. of The Carborundum Co. manufactures a backstand idler called the BS-4 in both standard and precision models. When extreme accuracies are required, the precision model is highly recommended. Rigidity of the mounting of the idler is also very important as any vibration set up in the idler could be transmitted through the abrasive belt and mark the workpiece. Care must also be taken to insure that the centerline of the idler pulley and the contact wheel form a right angle to the roll being ground (see Fig. 2.)

COOLANTS

Coolants are used primarily to maintain the roll at a constant and uniform temperature since localized heating can cause serious out-of-roundness and other inaccuracies. Thus, plenty of the proper coolant should be employed throughout the grinding and finishing cycle.

Coolants also carry away grinding swarf and particles of abrasive grain as the grain fractures to present new cutting points. In addition, it should contain enough detergent to coat the work with a thin rust-inhibiting film. Coolants which circulate through settling tanks should have an adequate filtering system to remove foreign particles such as sludge or gum. Good, efficient filters are necessary in any stage of roll grinding; but they are vital for high finishes. Coolants also aid in the prevention of belt loading, thereby insuring freer cutting action and longer belt life.

WORKPIECE HARDNESS

The material to be ground is also an important factor in the determination of feeds and speeds. Generally speaking, the harder the material to be ground, the slower the work speed and belt SFPM. Soft materials permit faster work and belt speeds. Soft materials also permit greater infeeds and more stock removal per hour. A desirable traverse rate is up to one half the belt width per revolution of the roll.

CONCLUSION

Roll grinding with abrasive belts has produced interesting results for many paper plants to date. New installations are being made constantly. Carborundum field representatives and engineers are well versed in the method, and can be invaluable to any organization anticipating the use of this new concept of roll grinding.

Beta Gages in Plastic Impregnation—A Case History

C. W. HANSEN

THIS case history describes the savings resulting from a product and process where the addition of beta ray gages and automatic control from gage information resulted in the reduction of product spread from ±25% to ±1%.

Radioactive isotopes have proved to be useful tools in industry for the measurement and control of many industrial processes. One of the first devices to use radioisotopes in industry is the beta gage, first developed by Tracerlab, Inc., of Waltham, Mass., which has been available as a commercial product for about 10 years.

The beta gage operates by absorption in the material to be measured of beta particles or electrons ejected from an unstable nucleus in its decay or transition to a stable form. Not all electrons escaping from a nucleus have the same velocity and some predictable percentage of them are absorbed by the material being gaged. The radiation which passes through the material is measured by a detector which is ordinarily an ionization chamber but which could be a Geiger, scintillation, or proportional counter. The ion chamber is used because of its stability, and the simplicity of the amplifier circuits used with it make it an ideal device for use under rugged industrial conditions.

Any form of inspection where a sample is taken from a process stream and checked, and the information used to correct the process, becomes part of a closed loop control system. A very stable process might be kept within tolerance by only an occasional check with a sample. However, very few processes are so stable that uniformity could not be improved by a continuous process check and control. The processes where a beta gage is the ideal measuring transducer are those which produce sheet materials such as paper, plastics, rubber, and foils as well as processes where these materials are processed by being coated, saturated, or laminated. The production from raw stock normally requires gaging at one point in the process but when a base material is being modified, a two-gage system is needed to obtain control information. The base stock weight and finished product weight are both measured and the former reading is subtracted from the latter automatically to obtain the added weight and the control information. The signal for control is a deviation from a set voltage and the phase and magnitude contain the information needed for process correction. There are ordinarily several elements of a process machine which can be used for control. These are such things as over-all machine speed, roll clearance, roll position, air flow, air pressure, fluid flow, fluid pressure, and temperature. The process will depend upon one of these factors as an adjustment to maintain uniform quality and the operator will have some method of introducing a change to correct for process variations.

These can all be grouped in two types of control depending on how they are operated. These are referred to as floating or nonfloating control. An example of a floating control is the screw down motor that positions a roll on a calender. It is powered for a time period proportional to the correction or position change required. After the power is removed it retains the setting until another correction is required. An example of a nonfloating control is a squeeze roll which obtains its force from a pneumatic cylinder. A change requires that the original pressure be modified up or down depending on the sign of the correction and that it be maintained at the new setting. Both types are found but the equipment to be described later is of the nonfloating type and uses a pressure cylinder on a squeeze roll. Automatic controllers can work with either type with equal accuracy.

C. W. HANSEN, Chief Engineer, Industrial Div., Tracerlab, Inc., Waltham, Mass.

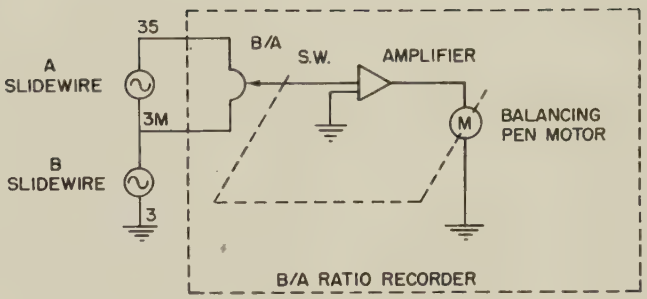

Fig. 1. Simplified ratio circuit

A problem of great significance in automatic control is the plant delay or time required for the product to travel from the control element to the gage. The automatic control must be programmed to wait for sufficient time after a correction to allow the corrected product to be gaged before another correction is made. Corrections made before this time will cause the product to oscillate in thickness and too long a delay will allow an excess of off target material to be processed. It can readily be seen that this is a critical factor in a good automatic control system.

The product to be measured and controlled in this instance is a plastic impregnated paper used in corrosive chemical applications. The base paper is pure cellulose with no clay fillers and it is saturated with resins to 200% weight. The paper stock is fed from a roll through beta gage A to obtain the base weight of the paper. It then goes through a saturating tank, air pressure controlled squeeze rolls and through the second beta gage B to obtain the wet coated weight. After entering the drying oven it goes through the third beta gage C to obtain the dry weight. Two control signals are obtained from the gaging system. These are B/A which is per cent saturation and B-A which is plastic weight.

The sheet width in this case is 24 in. so that the gaging equipment on the line is quite small. Larger sheet widths can be controlled as easily. It was felt desirable to obtain control information from the average weight of as much of the sheet as possible. Since a standing beta gage measures only a 2-in. circle, to obtain a reading that is equal to the average weight across the sheet many readings are taken by mechanically traversing the gage. The gage time constant is about 5 sec. so that by scanning the gage across the sheet in $^1/_{10}$ this time, a reading is obtained which is the average of all material scanned. The gaging head is supported on a track so that it can be moved in a reciprocating motion across the sheet by a pneumatic cylinder. It is recognized that this does not produce uniform velocity but the deviation from a true average reading caused by this is negligible. No effort was made to synchronize the motion of the three gages because the travel time was much faster than the gage time constant.

The gaging system used on this application is made to standardize automatically every half hour. To do this a timer in the gage console programs the system so that the gages are all withdrawn from the sheet, and the automatic controls locked at the last setting. Servo motors then drive potentiometers to compensate for electrical drifts, buildup of material on the source and detector windows, and source decay. The gages are then driven back on the sheet to again start cycling and the system is once more in control.

Each gage is calibrated to read the average weight of material passing through it in weight per unit area. These readings are presented on strip chart recorders and in order to obtain the ratio and difference signals, slave slidewires are connected to the recorder pen drive. In order to obtain the A/B ratio this circuit is used (see Fig. 1).

Slidewires A and B can be considered as generators with voltages proportional to the weight reading. Slidewire R, the amplifier and servo motor are in the recorder which shows the per cent saturation. If either A or B change, the input

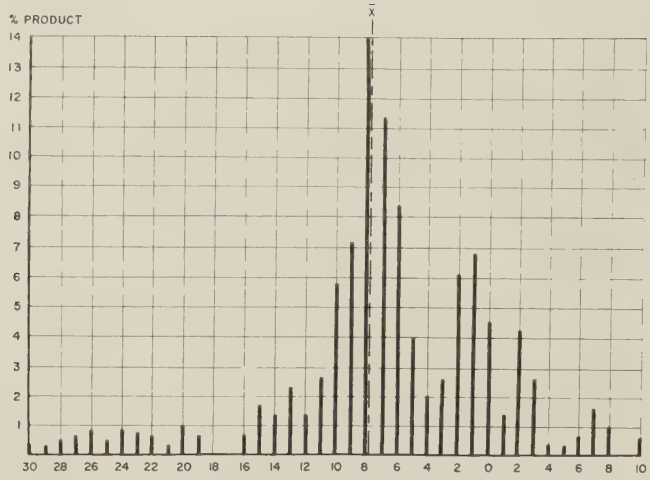

Fig. 2. Deviation from target saturation, %

to the amplifier will change and slidewire R will be driven to a new balance point. Changing A and B in the same ratio will result in zero change to the amplifier input and the recorder will not move.

The B/A control signal is obtained from a slave slidewire in the ratio recorder which is arranged in a bridge circuit with potentiometer used to set the operating point. The control used is a Leeds and Northrop series 60 proportional control. The output current of this unit is used to control an L & N electro-pneumatic converter and this in turn controls a pressure amplifier to get to the pressure needed to operate the squeeze rolls.

The B-A or plastic weight is also obtained from slave slidewires on the B and A recorders. This signal is also put into an automatic control but is not used in a closed loop control. The signal is used to vary the process speed in proportion to the amount of resin pickup. To maintain a set percentage of saturation the resin is increased as the base stock weight increases. This means that more heat will be required to cure it or that the material must remain in the oven for a longer time to have uniform properties. This is accomplished by making process speed proportioned to resin pickup. This is not a critical control factor so the loop is left open with no feedback from gage C. The time through the oven is quite long so that obtaining control information from gage C would have meant long delays. Gage C is for product information only and checks the operation of the system.

In order to establish the cost savings made possible by an installation of this sort it is necessary to perform an in-plant evaluation. The procedure for this is as soon as the installation is complete but before it is turned over to the machine operators, the gages are turned on and recording without any gage information being made available to the operators. This establishes the quality of product before any gage or control assistance.

The next step is to make the gage information available to the operator for manual control to see what is the best that can be done by a rapid feedback of information to the operator instead of requiring hand samples. The last step is to put the system on automatic control and again to collect data on product quality.

This evaluation was done without the second part because the unit was to perform automatic control and the customer was not interested in gage assisted manual control.

The first part of the evaluation showed that the average product or $\bar{x}$ was nearly 8% below the target saturation desired and that the total product spread was ±25%. The bar graph or histogram showing distribution is given in Fig. 2.

This shows the tabulation of all samples collected during this part of the evaluation.

The second part of the evaluation was with the gaging system in control. The $\bar{x}$ value was moved to a +0.1% and the product spread narrowed to ±1%. The chart shows a comparison of the two methods of operation (see Fig. 3).

This reduction of product spread makes available to the gage user a very substantial saving. Let us first assume that all of the material processed was all satisfactory and sold. This then would permit lowering the target to a point 24% below the present point with no material lighter in saturation than before. This would result in a 24% saving in resin since the product is sold by unit area and would also result in a saving on shipping costs. This may be more of a reduction in resin than the manufacturer wishes to make because he would like to raise his standards on minimum saturation. It is now possible for him to pick any operating point he desires and achieve savings and product improvement to whatever standards he sets. The addition of beta gages and automatic control to this process has given the customer the following benefits: savings because of scrap reduction, savings because of resin reduction, savings by reducing the number of hand samples which take time and material, and product uniformity impossible to achieve by manual control.

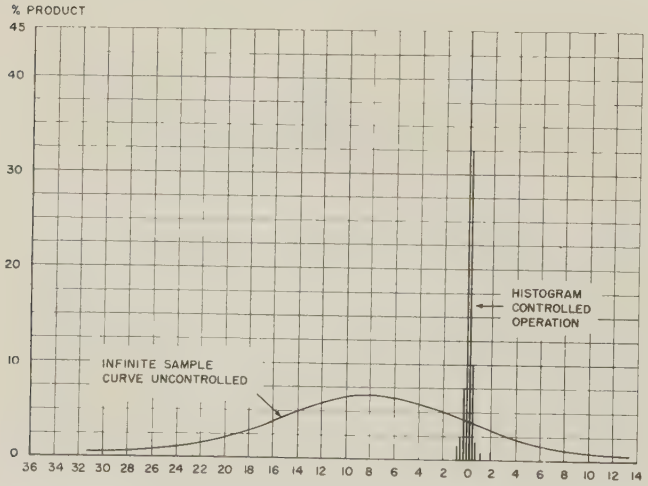

Fig. 3

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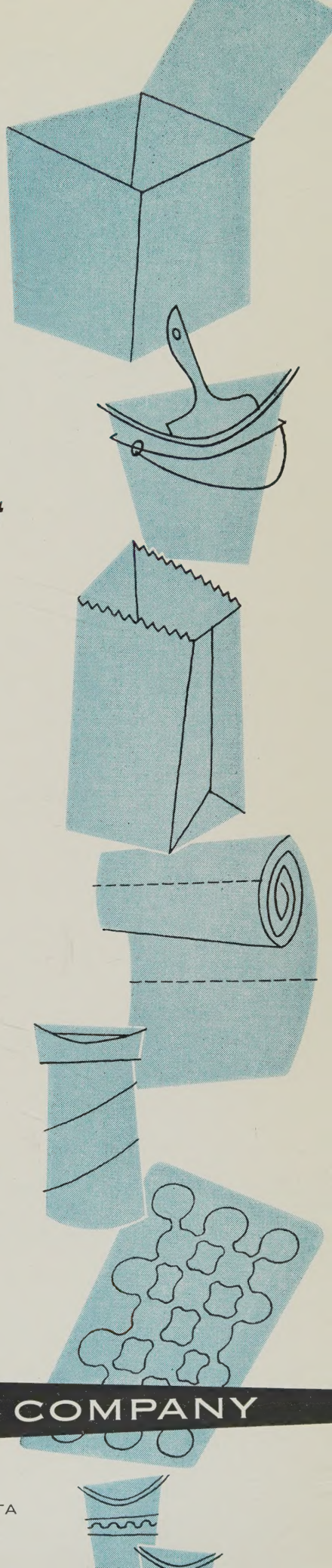

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